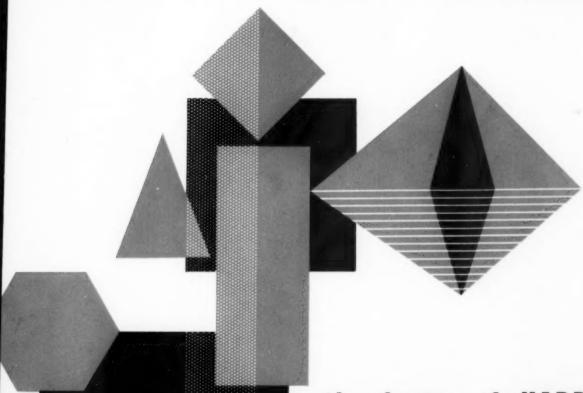
# TALLURGIA

THE BRITISH JOURNAL OF METALS

Vol. 64 No. 382

AUGUST, 1961

Monthly: Two Shillings and Sixpence



the factor of HARDNESS...



When your products are destined for years of hard labour, when hardness is a vital factor in their ability to withstand the wear and tear of a really hard life, then you need to adopt a hard and fast rule - always use Efco heat treatment furnaces.

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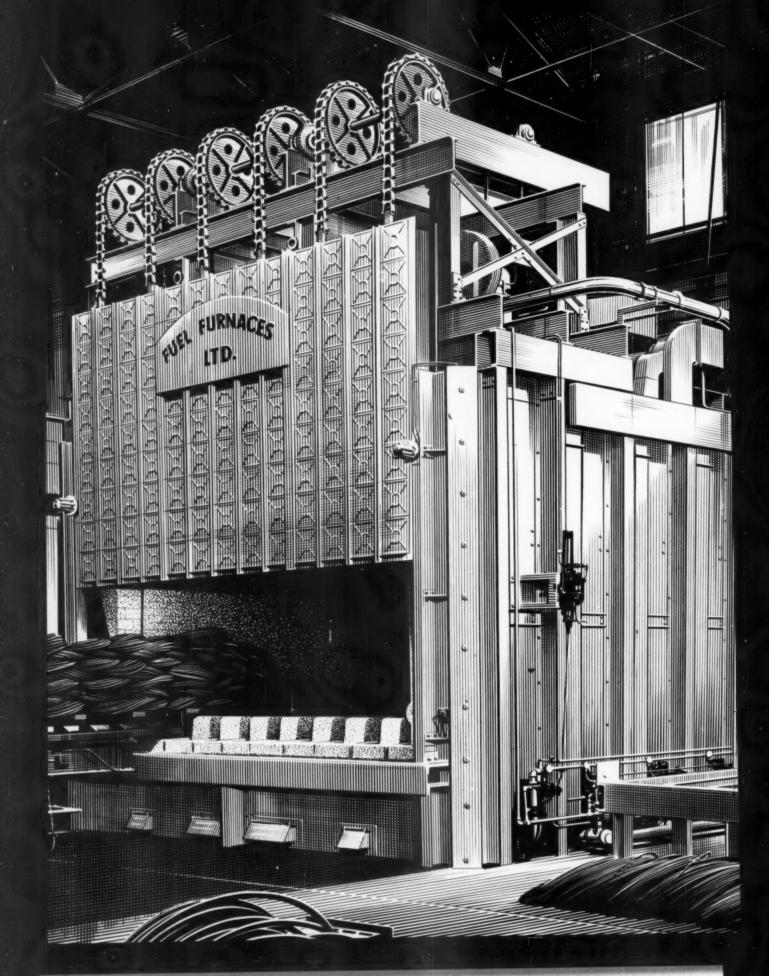
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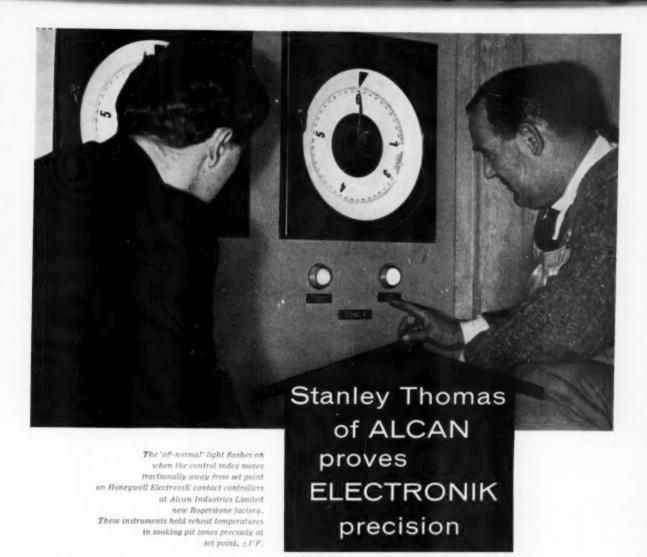
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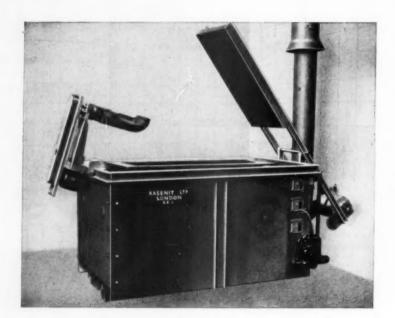


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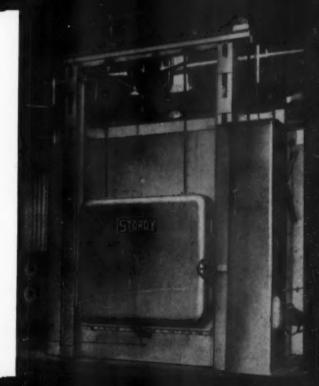


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Illustration: Electrically heated, air circulated batch-type furnace for aluminium. Working temperature 600°C. Heated length 20'6°.



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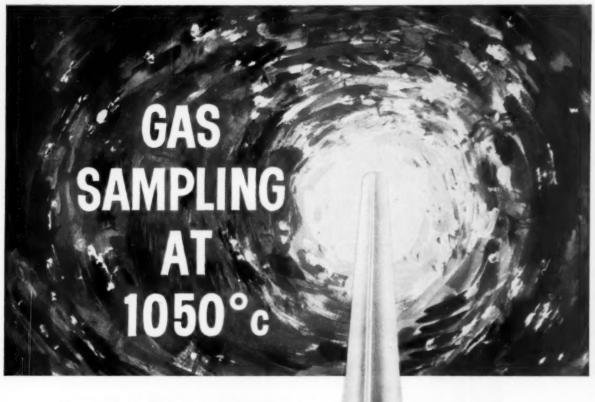
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Alumina	***	***	***	***	***	83%
Refractoriness - 5	Seger	Co	ne	-38	-	1850 C
Refractoriness und	ler l	oad				
28 lb./in.2 (2kg./cm.2	) -5	% de	efor	matio	n at	1660°C
After Contraction	-2	hrs.	at :-	_		
1600 C	***		***	***	***	0.40%
<b>Apparent Porosity</b>			***	***	***	23%
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	13,0	00 lb	./in.	2 (91	4 kg	/cm.2)

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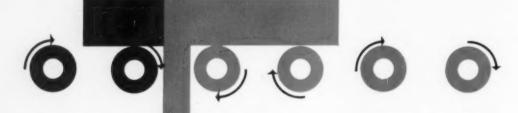
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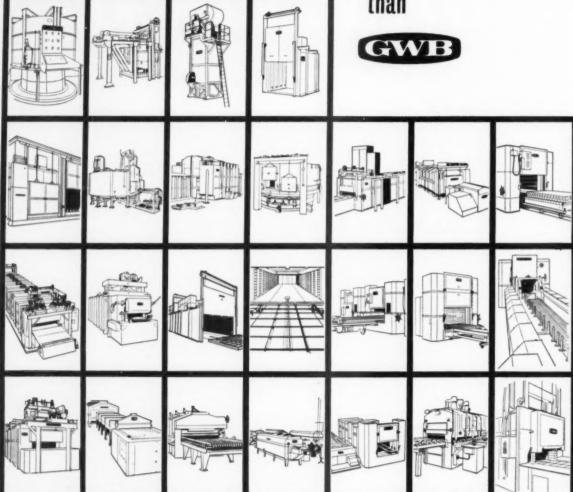
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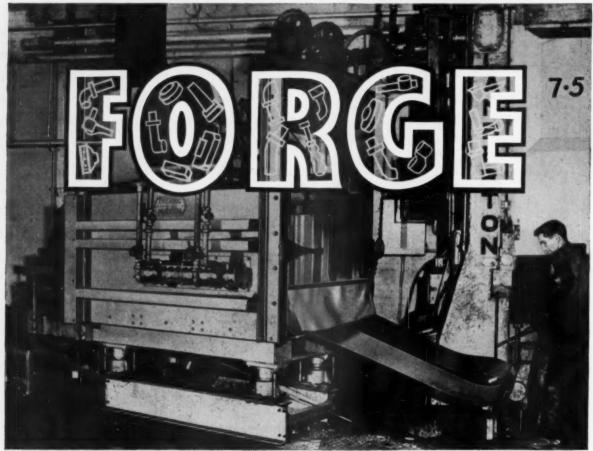
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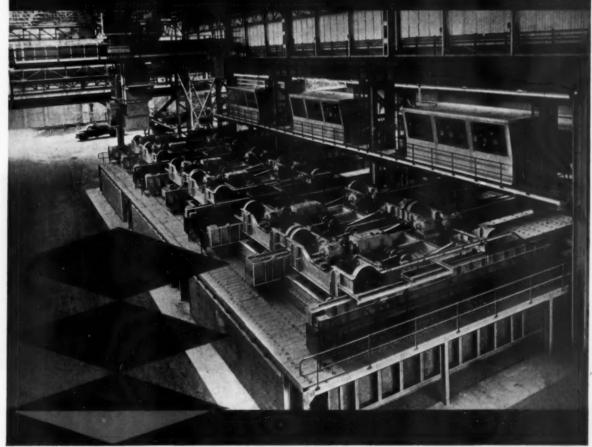
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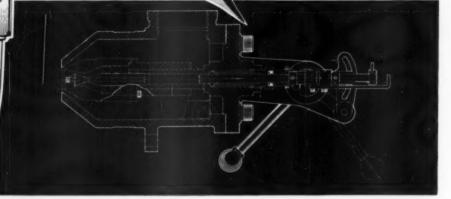
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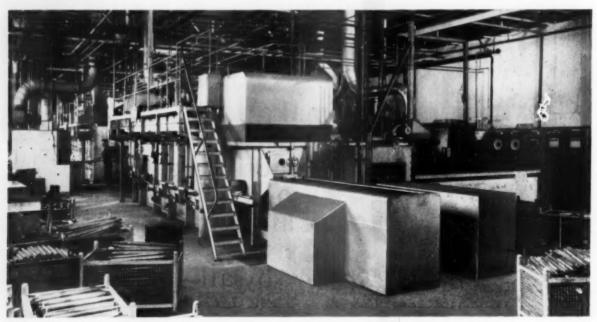
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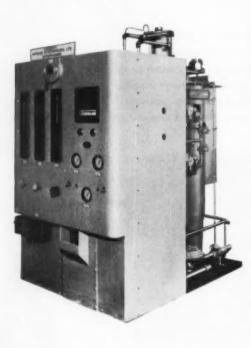
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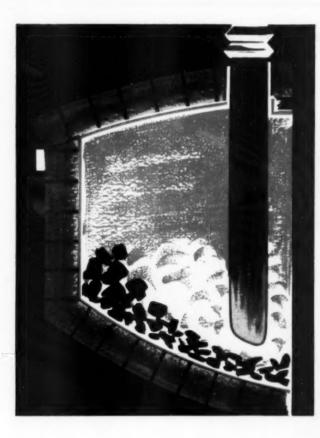
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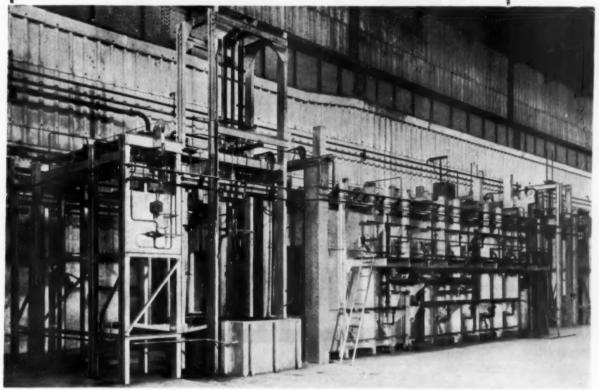
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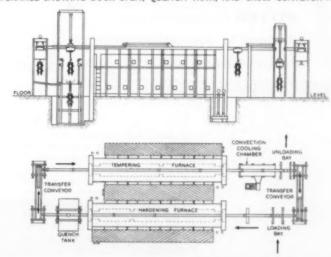
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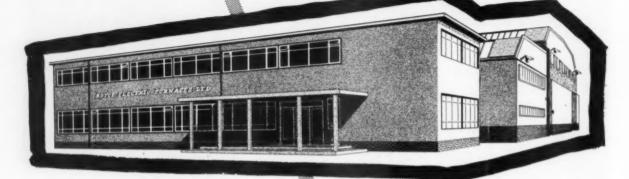
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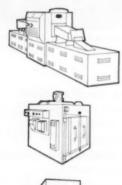


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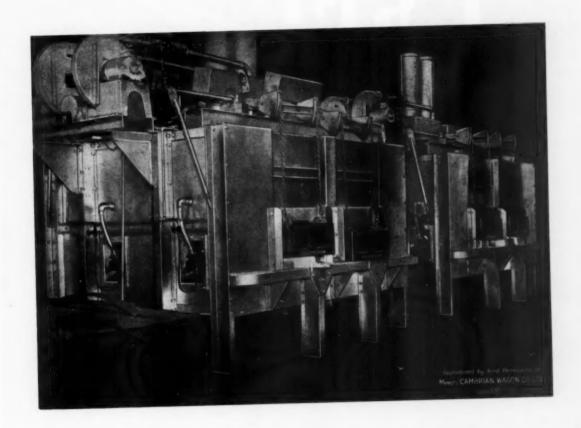
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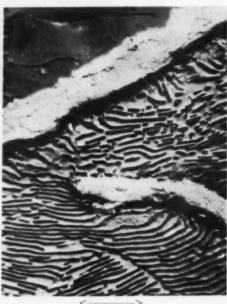
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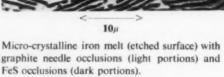
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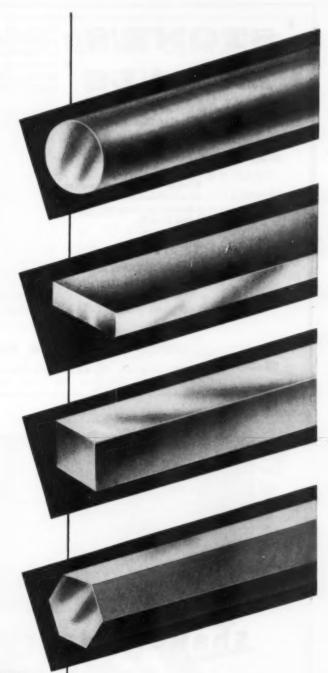
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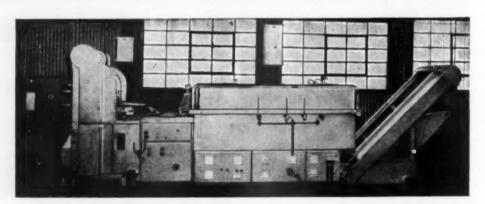
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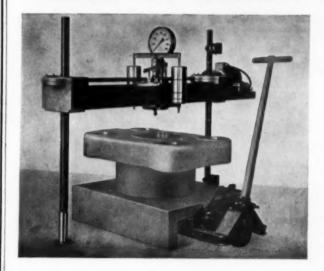
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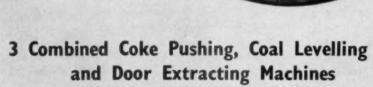
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# METALLURGIA

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#### Co-operative Industrial Research

THE Department of Scientific and Industrial Research like Heaven-helps those who help themselves as far as co-operative industrial research is concerned. and it is to be hoped that the promised or threatened (depending on point of view) cuts in Government expenditure are not achieved at the expense of the industrial research associations, which are playing an important role in the steady expansion of industrial research and development activities. The income for each association is made up of contributions from industry and grants from the Department, the latter being related to the needs of the association and the financial support the industry concerned is willing to give, so that in more than one sense the grant is earned. The total income from industry and Government, which rose from £6.9 million in 1958 to £7.3 million in 1959. has now reached £7.9 million, the Department's grant having risen to £1.8 million. This is in part—but by no means entirely—due to the introduction of two new associations to the Government scheme, bringing the number of grant-aided research organisations up to

In its recent annual report\* the Industrial Grants Committee of the Council for Scientific and Industrial Research expressed concern at the rising cost of research over past years and at the requirement for income progressively to increase, if the level of research undertaken in any of the associations is to be maintained in real terms. This problem affects all the research associations, but it is a critical one in the case of the associations serving small industries. What was adequate to support a small but active research group ten years ago is no longer so, and some of the smaller organisations have been experiencing difficulty in raising adequate grantearning industrial income.

It is impossible to specify a minimum expenditure level for a co-operative effort to be profitable in the research field but, in the opinion of the committee, it is essential that a research association's income should be adequate to meet the technical needs of the industry, and at the same time to offer an attractive prospect to new young research workers—both requirements being regarded as essential for a healthy research association. The size of an industry is not, of course, the sole criterion of its economic or technical importance, since some industries exploit valuable overseas markets and others specialise in the manufacture of the components used in other more complex products.

The problem facing the Industrial Grants Committee is that of determining in particular cases whether it is reasonable to expect industrial support to grow to a point where a well-equipped though small research body can be maintained on a long-term basis. Grant-aid is normally given on the basis of a continuing long-term programme which, it has always been implied, expects the support of continuing partnership between Government and the industry concerned. But, warns the Committee, the Department of Scientific and Industrial Research must be satisfied that co-operative schemes are technically and economically sound before offers of grant are made. Even where grant-aid is given, the research associations may experience recurring difficulties due to inadequate industrial support or doubtful future profitability, and in such cases it is D.S.I.R.'s duty to consider whether grant-aid should be continued, or whether some alternative arrangements might not meet the industry's needs more effectively.

One possibility in this respect is some form of cooperation between research associations. Quite often it would be possible for a larger kindred organisation to afford valuable facilities, but such help should not discourage the smaller industry from a full acceptance of its responsibilities in providing an adequate level of financial support. This is no tentative suggestion: it has been put to the test, and today the File and Cutlery Research Councils occupy part of the Sheffield laboratories of the British Iron and Steel Research Association. Nor is this the first instance, for the Heating and Ventilating Research Association spent its early years in similar circumstances, as did the Coil Spring Federation Research Organisation. The latter, like the File and Cutlery Research Councils, enjoyed the hospitality of B.I.S.R.A.'s Sheffield laboratories, and now that the Organisation has its own premises next to B.I.S.R.A. there is still a fair amount of co-operation between the

The Committee has considered other possible ways of meeting the research needs of small industries: by placing contracts with established research organisations, or even through central facilities for industries whose problems would not of themselves justify a permanent independent laboratory.

The overall problem remains and will continue to receive attention, but the Industrial Grants Committee, points to the small cutlery industry as one which has faced up to this problem of rising costs and placed the Cutlery Research Council on a sounder financial basis. The Council first received grant-aid in 1951, and the bulk of the industrial income has since been in the form of an annual block contribution from the Sheffield Cutlery Manufacturers Association. This contribution had remained almost static since 1951, but arrangements have now been made through the Board of Trade for future industrial income in support of the Council's research programme to be collected by a statutory levy.

It is D.S.I.R. policy to encourage research associations to recruit Commonwealth firms into membership, but no grant is paid in respect of income from them. Nor, to

<sup>6 &</sup>quot;Research for Industry 1960", H.M. Stationery Office.

date, has any grant been paid by Commonwealth Governments in respect of income from such firms, and negotiations are to be opened by the Department with each of these Governments with a view to obtaining agreement to their paying grants on the income received by U.K. research associations from Commonwealth member firms.

During the past year the Committee has reviewed its policy towards the admission of non-Commonwealth overseas firms to membership of research associations, and has recommended that the Department, with certain safeguards, should now permit research associations to admit foreign firms to membership where it appears advantageous for them to do so. These recommenda-

tions have been accepted by the Research Council and research associations have been invited to submit schemes for the admission of foreign firms, subject to certain conditions. The consent of D.S.I.R. must be given for the admission of such firms, who will not be allowed to vote at the annual general meeting, nor to be represented on the council of the association. Furthermore, all members of a research association should be given an opportunity of placing before their council any commercial reasons they may have for wishing to oppose the admission of a foreign firm. The financial benefit to the association will be confined to the firm's subscription, since no grant will be payable in respect of such income as may be attracted from this source.

#### New and Revised British Standards

METHODS FOR THE ANALYSIS AND TESTING OF COAL AND COKE: PART 16: REPORTING OF RESULTS (B.S.1016: PART 16: 1961) PRICE 88, 6d.

In parts 1–11 of B.S.1016, the sample from which the analytical results are obtained is the analysis sample, prepared in such a manner that replicate determinations can be made with a minimum of error. The results of determinations on this basis are, however, not of immediate commercial value, as they are not related to the coal or coke as-received, nor to the behaviour of the fuel in use. It is therefore necessary to convert such results to other bases, e.g. 'as-received' or 'dry, ash-free,' and Part 16 of B.S.1016 explains the procedures to be used for this purpose, whether for coal or coke.

Although the permitted tolerances for the standard methods described in B.S.1016 are stated, occasions may arise where the reliability of a set of results is in question, and Part 16 therefore includes a section outlining a suitable approach to this problem. An appendix has also been provided detailing the calculation from which the conversion factors used in Parts 1–11 have been derived.

METHODS FOR THE SAMPLING AND ANALYSIS OF TIN AND TIN ALLOYS: PARTS 3, 4, 5, 6 and 8 (B.S.3338: PARTS 3, 4, 5, 6 and 8). PRICE 3s. EACH PART.

FIVE more parts of B.S.3338, which lays down methods for the sampling and analysis of tin and tin alloys, have been published. They are: Part 3—Determination of antimony in ingot tin (photometric method); Part 4—Determination of copper in ingot tin and tin-lead solders (photometric method); Part 5—Determination of lead in ingot tin and tin-antimony solders (photometric method); Part 6—Determination of copper in high purity ingot tin (photometric method); Part 8—Determination of bismuth in ingot tin, tin-lead solders and white metal bearing alloys (photometric method).

The methods are intended for the sampling and analysis of the grades of metal covered by B.S.3252, "Ingot tin," B.S.218, "Soft solders" and B.S.3332, "White metal bearing alloy ingots." Part 3 deals with the determination of antimony in ingot tin having an antimony content up to 0.25%; Part 4 with the determination of copper in ingot tin and tin-lead solders, having a copper content from 0.01 to 0.4%; Part 5 with the determination of lead in ingot tin and tin-antimony solders, having a lead content up to 0.9%;

Part 6 with the determination of copper in high purity ingot tin, having a copper content up to 0.04%; and Part 8 with the determination of bismuth in ingot tin, tin-lead solders and white metal bearing alloys having a bismuth content of up to 0.12%. In each case, required reagents are specified and methods of sampling and test procedure are recommended.

Dental Cobalt Chromium Casting Alloy (B.S.3366: 1961). Price 3s.

This new British Standard specifies basic requirements for cobalt chromium casting alloy used for dentistry: It does not relate to surgical implants. Composition and mechanical properties are laid down, and any package of ingots claimed to comply with this standard must be marked with the name or trade mark of the supplier, the weight of the contents of the package, the batch number and the number of the standard. The B.S.I. Kitemark may also appear, if the manufacturer is licensed to use it. The presence of this mark on a product shows that B.S.I. confirms that the product is up to standard.

Dental Gold Solders (B.S.3384: 1961). Price 3s. REQUIREMENTS for two types of dental gold solders are laid down in this new British Standard: those intended for general purpose use (type A) and those of lower gold content, not generally recommended for fixed restorations (type B). Composition, shape and size, workmanship, melting range and quality of union are dealt with, and appropriate tests are laid down. When a solder is claimed to comply with the requirements of this standard. packages must be marked with the name or trade mark of the supplier, the type of solder (type A or type B), the solidus and liquidus temperatures, the weight, and the number of the standard. The B.S.I. Kite-mark may also appear, if the manufacturer is licensed to use it. The presence of this mark on a product shows that B.S.I. confirms the manufacturer's claim that the product is up to standard.

Copies of these Standards may be obtained from the British Standards Institution, Sales Branch, 2 Park Street, London W.1. Postage will be charged extra to non-subscribers.

The Birmingham office of British Driver-Harris Co., Ltd., has been moved from 55 George Street, Parade, Birmingham, 3, to Target Works, Hancock Road, Birmingham 8. Telephone: East 3761.

# An Investigation Into the Causes of Intercrystalline Corrosion in Zinc-Aluminium Alloys

By C. W. Roberts, B.Sc., Ph.D., F.I.M.\*

The results are presented of an investigation into the fundamental causes of the intercrustalline attack that occurs in high-purity zinc-aluminium alloys, and into the action of impurity or alloying elements in accelerating or retarding the corrosion. It is shown that susceptibility to this form of attack in the presence of moisture can be substantially counteracted by the addition of magnesium to the alloys, provided that the concentrations of impurities present are below the limits closely defined in the British Standard specification for commercial alloys.

N a paper published during 19511 the available literature on the subject of intercrystalline corrosion in zinc-aluminium alloys was reviewed and the results obtained from experimental work designed to elucidate certain aspects of the problem were reported. Whilst this work clarified a number of points, it did not provide a satisfactory explanation of the fundamental cause of the attack that occurs in high purity alloys, nor of the action of impurity or alloying elements in accelerating or retarding the corrosion. A further programme of research work was therefore undertaken with the object of discovering the fundamental cause of the corrosion and of evolving, if possible, means of rendering these alloys immune to this form of attack. The experimental methods used in this investigation and the results obtained are reported below.

In the interest of clarity this paper has been subdivided into sections, each section comprising a description of the experimental methods adopted, a summary of the results obtained, and brief comments on these results where appropiate. All results are correlated and discussed in the final section, but, to maintain continuity, certain results and ideas are discussed at the beginning

of the relevant section.

The materials used for the preparation of the alloys examined were of the following grades:

Zinc: Special Purity (S.P.) Zinc: Crown Special (C.S.) Purity 99-999% Purity 99.99% Purity 99.98% Aluminium Purity 99 - 95% Copper Other Metals Purest grade available

#### I. The Effect of Time, Atmosphere and Temperature on Rate of Intercrystalline Attack

#### (a) Effect of Time

Previous work had shown that zinc-aluminium alloys of composition within the range 0.02-22% aluminium are susceptible to intercrystalline attack when exposed to an air/water vapour atmosphere at 95° C .- the test conditions normally used by previous investigators. To assess the effect of time of exposure in such an atmosphere on the extent of the attack, three alloys designated A. B and C, containing, respectively, 0.10%, 4.0% and 20% aluminium, were prepared, the zinc being of purity 99.999% and the aluminium 99.98%. These particular compositions were selected because A is of the highest

TABLE 1.—EFFECT OF TIME ON DEPTH OF INTERCRYSTALLINE ATTACK ON THREE ZING-ALCMINIUM ALLOYS.

Time of	Depth of Penetration of Intercrystalline Attack (in.)			
(days)	Alloy A (maximum)	Alloy B (average)	Alloy C (average	
1	0.003	0.002	0.001	
2	0.007	0.0015	0.002	
3	0-009	0.004	0.005	
4	0.020	0.004	0.005	
5	0-024	0.005	0.007	
6	0-024	0.005	0.007	
7	0.030	0-007	-	
8	0-032	_	0.014	
90	0.045	0.008	0.014	
10	0.050	0.011	0.012	

aluminium content with which an alloy can safely be assumed to be single phase at 95° C., B is of the aluminium content normally used for commercial alloys, whilst C is of a composition near to that of the "eutectoid alloy, which composition has been shown to be very susceptible to severe attack.

Specimens of each alloy were exposed to an air/water vapour atmosphere at 95° C. for periods of time from I to 10 days, the depth and severity of the resulting intercrystalline attack being assessed subsequently by metallographic methods. The figures obtained for depth of penetration are shown in Table I and the type of corrosion observed is illustrated in Figs. 1, 2 and 3.

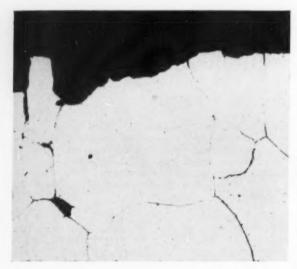
These results show that the relationship between time of exposure and depth of penetration in all three alloys is approximately linear, although there is a fair degree of scatter in the results obtained from Alloy C. This finding is interesting, since it indicates that there is no tendency for attack to "stifle" itself with time.

#### (b) Effect of Atmosphere

Since the atmosphere normally used to assess the susceptibility of zinc-aluminium alloys to intercrystalline attack is a mixture of air and water vapour, it appeared desirable to examine the effect of variations in the ratio of nitrogen, oxygen, carbon dioxide and water vapour on the rate of corrosion. However, as it is well established that no grain boundary reaction occurs when these alloys are heated in dry air at all temperatures, it seemed that the first point to examine was the effect of water vapour

For this purpose, specimens of each of the Alloys A, B and C were sealed in glass tubes containing a small quantity of distilled water. Before sealing, the tubes

<sup>&</sup>lt;sup>6</sup> Metallurgical Consultant. Formerly Group Leader, Extraction Metallurgy, Research Department, Imperial Smeiting Corporation, Ltd.



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Fig. 1.—Intercrystalline corrosion in the alloy containing 0.10% aluminium. Unetched. ×500

were evacuated, the contained water being frozen prior to this operation. The sealed tubes were suspended in a steam tank alongside a similar set of specimens and all were held at a temperature of 95° C. for ten days, the second set, of course, being exposed to a mixed air/water-vapour atmosphere.

The results obtained from the subsequent metallographic examination are shown in Table II and appear to indicate that the corrosion is brought about by water vapour alone, the simultaneous presence of air having no noticeable effect. In view of this finding there appeared to be little point in examining the effect, if any, of variations in the nitrogen, oxygen and carbon dioxide concentrations in the atmosphere.

#### (c) Effect of Temperature

Since it was proposed to examine the effects of temperature to a maximum of about 150° C., a thermostatically controlled autoclave was used for these tests, and to avoid any possibility of contamination by jointing compounds, rust etc., the test specimens, which were of Alloys A, B and C, were sealed in glass tubes containing water alone, as in the tests described in (b) above. The pressure exerted by the water vapour within the glass tubes at temperatures above 100° C, was balanced by the pressure generated by the water contained in the autoclave itself. It was assumed that the rate of corrosion

TABLE IL—EFFECT OF ATMOSPHERE ON DEPTH OF INTER-CRYSTALLINE ATTACK ON THREE ZINC-ALUMINIUM ALLOYS.

Condition	Depth of Penetration of Intercrystalline Attack (in.)				
of Test	Alloy A (maximum)	Alloy B (average)	Alloy B (maximum)	Alloy C (average)	
At 95°C, in steam tank (with access of air)	0-050	0.011	0.030	0-012	
At 95°C, scaled in glass tube over water (air ex- cluded)	0-070	0.013	0.025	0.011	

TABLE III.—EFFECT OF TEMPERATURE ON INTERCRYSTALLINE

Temperature (° C.)	Duration		of in./day)	
	of Test (days)	Alloy A (maximum)	Alloy B (average)	Alloy C (average
80 95 106 116 130 149 155	14 10 2 2 14 1	0-0017 0-007 0-0175 0-0225 0-038 0-120 0-170	0-0006 0-0013 0-0025 0-007 0-0136 0-017	0·0004 0·0011 0·004 0·007 0·012 0·019 0·025

would increase with increasing temperature, and the duration of the tests at the higher temperatures was decreased to keep the depth of the corroded layer within reasonable limits.

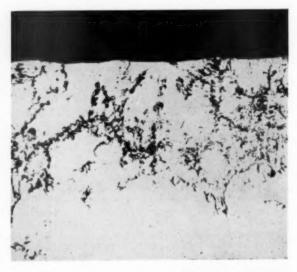
The subsequent examination was carried out by conventional metallographic methods, the results obtained being shown in Table III. The plot of the logarithm of the figures for rate of attack (penetration/time)  $v.\ 1/T$  approximates to a straight line in all three cases; the relationship between rate of corrosion and temperature of corroding medium can therefore be conveniently expressed by the equation:

$$\log d = a - \frac{K}{T}$$

where d is the rate of corrosion, T the absolute temperature, and a and K constants.

#### Comments on Results of Section I

The results obtained from the tests described are, for the most part, as might have been predicted from the generally accepted theory of corrosion. However, the fact that the alloys are corroded by water vapour in the absence of air does not appear to have been noted by other investigators and, since air alone does not cause preferential attack at grain boundaries, this finding appears to lend support to the view that the corrosion



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Fig. 2.—Type of intercrystalline attack observed in the alloy containing 4.0% aluminium. Unetched. ×500

is electrochemical in origin—assuming that under the conditions of test the water vapour was "wet." The next series of experiments was therefore designed to determine whether the corrosion was, in fact, brought about by electrochemical reaction.

#### II. Corrosion by Dry Water Vapour

It appeared that the most convenient method of determining whether or not the reactions that brought about intercrystalline corrosion in these alloys were electrochemical in origin was to expose specimens in an atmosphere of "dry" steam (where electrochemical reaction cannot, presumably, occur) at a temperature similar to that at which "wet" steam tests on corresponding alloys had been carried out, and to compare the corresponding degrees of corrosion.

For this purpose, a cylindrical electric furnace was mounted in a vertical position and fitted with an internal copper coil which occupied the lower half of the furnace tube and which was connected to a water boiler situated below the furnace. Both ends of the furnace tube were closed with rubber bungs, the upper carrying the controlling thermocouple, a thermometer and a steam outlet tube, this outlet tube being connected to an external condenser. In operation, the specimens were suspended at the mid point of the furnace (which corresponded with the upper end of the superheating coil), the temperature of the furnace was adjusted to 150° C. and steam from the boiler was allowed to flow. The steam, which was superheated to 150° C. in the copper coil. impinged on to the specimens and then passed into an external condenser, the water produced then returning to the boiler. As the condenser was open to atmosphere. the pressure of the system was atmospheric and hence the steam was " dry.

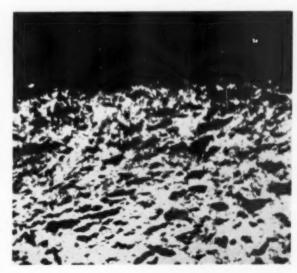
Specimens of alloys of identity number and composition shown in Table IV were exposed in this apparatus for a period of five days and the extent of the resulting intercrystalline attack was assessed by metallographic means. The results obtained are also shown in this table.

The conclusions that emerge from these results are :-

- Intercrystalline corrosion will occur in single phase zinc-aluminium alloys when exposed to dry water vapour, i.e. in conditions where electrochemical action is not possible.
- (2) The presence of magnesium in the alloy reduces the susceptibility to intercrystalline attack.

TABLE IV.—EFFECT OF ADDITIONAL ELEMENTS ON THE INTERCRYSTALLINE ATTACK IN A ZINC-0-10% ALUMINIUM ALLOY EXPOSED TO DRY WATER VAPOUR AT 150° C.

Alloy No.	Composition	Type of Intercrystalline Attack and Depth of Penetration
4/1	C.S. Zinc+0·10° Al	Wide spines to a maximum depth of 0.050 in,
4/2	C.S. Zinc+0·10% Al +0·005% Mg	Very infrequent spines to a maximum depth of 0.020 in.
4/3	C.8. Zinc+0·10% Al +0·020% Mg	Spines occurring more frequently than in Alloy 4/2 to a maximum depth of 0.015 in.
4/4	C.8. Zinc+0·10% AI +0·30% Cu	Spine-type corrosion, considerably less severe than in Alloy 4/1, to a maximum depth of 0-055 in.
4/5	C.S. Zinc+0·10% Al +0·02% Pb	Fairly severe spine-type corrosion through specimen, i.e. depth of penetration greater than 0-125 in.



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Fig. 3.—Type of intercrystalline attack observed in alloy containing 22 0% aluminium. Unetched.  $\times\,500$ 

(3) The presence of lead in the alloy increases the rate of attack markedly.

The rate of penetration of intercrystalline attack in single phase alloys when exposed to wet steam at  $150^{\circ}$  C. was found to be  $0\cdot120$  in./day (Table III), whereas in dry steam the rate was  $0\cdot010$  in./day. The tests in dry steam were carried out at atmospheric pressure, while in the wet steam tests the pressure increased with increasing temperature, being  $66\cdot7$  lb./sq. in. at  $150^{\circ}$  C. It is not possible to assess the effect of change in gas pressure on the rate at which a solid/gas reaction proceeds unless the rate-controlling process can be identified (whether, for example, it is diffusion controlled). In the present case it cannot be identified, but it is unlikely that an increase in gas pressure from  $14\cdot7$  (atmospheric) to  $66\cdot7$  lb./sq. in. will increase the rate of reaction by a factor of more than about 2.

It appears justifiable to conclude, therefore, that while the intercrystalline attack is due fundamentally to a chemical reaction between water and the alloy (or one component of the alloy), the rate of the reaction, and consequently the rate of corrosion, is increased markedly (by a factor of 6 or more at  $150^{\circ}$  C.) when electrochemical reaction is also possible. The question of whether or not electrochemical action does occur will be discussed later.

#### III. The Chemistry of the Grain Boundary Reaction

Since high purity zinc (99·999%) and, indeed, zinc containing appreciable quantities of impurities such as lead, tin, and cadmium, is not susceptible to intercrystalline attack when exposed to water vapour whilst zinc-aluminium alloys are, it must be assumed that the presence of aluminium, even when completely in solid solution in the zinc crystals, increases the reactivity of the grain boundaries of these alloys.

Aluminium, as an element, is, of course, very reactive in the presence of water and is strongly anodic to zinc, but under normal conditions the metal is protected by

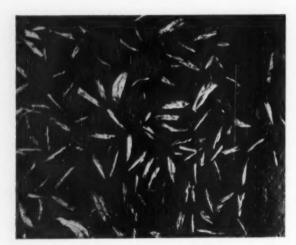


Fig. 4.—Crystals from a specimen of an alloy that had suffered severe intercrystalline attack.  $\times 2\frac{1}{2}$  approx.

a tightly adhering oxide film and these characteristics are therefore masked. Taking these facts into consideration in relation to the structure of a single phase zine-aluminium alloy, it appeared that the grain boundaries of such an alloy would be more reactive than the grain interior if one or more of the following conditions obtained:—

- (a) the aluminium was at a higher concentration in the zones of the crystals adjacent to the grain boundaries;
- (b) the aluminium atoms within the grain boundary zones were more mobile than those within the crystal: or
- (c) a protective film of corrosion product could not form at the point of attack,

That the solute in single phase alloys may segregate at the crystal boundary zones without precipitation occurring has been shown by a number of recent investigations, the phenomenon being described as "equilibrium segregation" 2–8.

That solute atoms may be more mobile at grain boundary zones than within the crystal appears to be implied by the fact that in many supersaturated alloys precipitation occurs more readily in these zones than within the crystal.

If equilibrium segregation of aluminium in these single phase alloys does occur, or if the aluminium atoms are more mobile at the grain boundary zone, preferential oxidation of aluminium will take place during intercrystalline attack by water vapour. To examine this point an alloy of composition C.S. Zine  $+~0\cdot10\%$ aluminium + 0.05% lead was prepared, the lead being added to increase the rate of attack. Specimens of the alloy were exposed to steam for a period of time sufficient to allow the intercrystalline attack to penetrate the metal completely. On application of slight pressure the specimens disintegrated into small separate crystals, the approximate average dimensions being  $3 \times 1 \times 0.5$  mm. The crystals were then vibrated vigorously to detach the corrosion product, which was separated by sieving through a 240 mesh screen. The alloy crystals, some of which are shown in Fig. 4, were spread over a flat surface

and a few grams were separated with the aid of a hand lens and forceps, care being taken to ensure that each unit selected was a single crystal and not an agglomerate (with corrosion product included). One half of the sample thus obtained was shaken vigorously with water to remove any corrosion product still adhering and the other half was washed for a few seconds in 1% hydrochloric acid for the same purpose. The respective aluminium contents of the two samples thus prepared and that of the original alloy were determined by chemical analysis, the results obtained being as follows:

Original	alloy .			 0.098% Al
Corroded	crystal	(water washed	)	 0.082% Al
Corroded		(acid washed)		 0.085% Al
		Accuracy . O. l	1050/	

The corrosion product (-240 mesh material) was examined by X-ray diffraction, the pattern obtained being that of metallic zine with faint indications of the presence of zine oxide. To examine the material in greater detail the sample was heated at 450° C. under vacuum in a Pyrex glass vessel, the zine vapour evolved being condensed on a cold finger. From the respective weights of the components it was estimated that the original material (-240 mesh) contained about 33% metallic zine and (presumably) 7% combined water.

The residue from this distillation was also examined by X-ray diffraction, the pattern obtained indicating the presence of zinc oxide only. The material was then analysed spectrographically, the standards used being intimate mixtures of zine oxide and aluminium oxide; it was estimated that the aluminium content of this material was 0·24%. The average aluminium content of the original material (60% mixed oxides, 33% metallic zine and 7% water) was therefore 0·14%. For the purpose of comparison it was calculated that if a homogeneous alloy containing 0·10% aluminium corroded evenly to yield a corrosion product 60% mixed oxides, 33% metallic zine and 7% water, the aluminium content of the material would be 0·081%.

To obtain confirmation of the results obtained from the experiments described above, the specimen of Alloy No. 4/5, which was exposed in dry steam at a temperature of  $150^{\circ}$  C., was separated into single crystals by the same method and the crystals (after washing in 1% hydrochloric acid) together with the original alloy, were analysed chemically. The results obtained were:

Original alloy . . . . . . 
$$0.095\%$$
 Al Corroded crystals . . . . .  $0.085\%$  Al Accuracy  $\pm 0.005\%$ 

These results clearly indicate that during the intercrystalline attack the aluminium contained in the single phase alloy is oxidised preferentially, and the possible reasons for this preferential attack in an ostensibly homogeneous alloy were next examined.

#### IV. The Electrochemistry of the Corrosion Process\*

On the assumption that the increased reactivity of the grain boundary zones in single phase zinc-aluminium alloys is due to the occurrence of equilibrium segregation, possible methods of determining whether or not this occurred were considered. The most effective method of detecting equilibrium segregation of the solute in a

<sup>\*</sup> The writer is indebted to Dr. U. R. Evans for valuable guidance on this aspect of the investigation. Dr. Evans does not necessarily agree, however, with all or any of the views expressed or the deductions made.

TABLE V.-POTENTIALS AND CURRENT AFTER 3 HOURS AND 15 HOURS

4 D	Composition	Period of	Potential on Saturated Calomel Scale (V.)		Current
Alloy No.	Composition	Immer- sion (hr.)	Grain Boundaries	Grain Interiors	(mA.)
5/1	C.S. Zine	3 15	-0.950 -0.960	-1.000 -1.009	0·25 0·40
5/2	C.8. Zinc+0·10% Al	3 15	-0.994 -1.001	-1:005 -1:011	0.08
5/3	C.S. Zinc +0·10% Al +0·30% Cu	3 15	-0.972 -0.966	- 0-969 - 0-967	0.01
5/4	C.S. Zinc +0·10% Al +0·01% Mg	3 15	- 0 · 983 - 0 · 980	0 · 990 1 · 002	0·03 0·13

single phase alloy appears to be that used by Chalmers and his associates.\* Enquiries revealed, however,† that the radioactive isotopes of aluminium are very unstable. the half-life being about three minutes. The preparation of an alloy from this material is therefore not practicable.

With regard to electrochemical methods. Mears and Brown used such a method to show that when aluminium-copper alloys are in the "susceptible state" (with respect to intercrystalline attack) less copper is in solution in the zones adjacent to the grain boundaries than in the remainder of the crystal, thus rendering the zone anodic to the interior. Further, Perryman<sup>6</sup> adopted the method developed by these investigators to demonstrate the occurrence of equilibrium segregation in R-brass

In the case of single phase zinc-aluminium alloys, it appeared reasonable to assume that if aluminium were present in the grain boundary zone at a higher concentration than in the interior zones of the grain, the respective solution potentials of these zones would be altered by different amounts compared with pure zinc.

It was decided to proceed with the electrochemical method and the technique developed by Mears and Brown was selected in preference to that involving the use of a probe electrode extending into a capillary, since, by the former technique, current flow can be measured in addition to solution potentials.

#### Experimental Details

Alloys of the composition shown in Table V were prepared and cast into bars of convenient dimensions.

† The writer is indebted to Mr. J. L. Putman of A.E.R.E., Harwell, for this

Alloys Nos. 5/3 and 5/4 were included since it appeared possible that the presence of copper or magnesium in an aluminium-containing alloy might prevent the occurrence of equilibrium segregation—assuming that this occurred in alloys free from these two elements. The bars from each alloy were, in sequence, remelted in glass tubes, If in, in diameter, and, after stirring, the tubes were lowered slowly through a vertical furnace with the object of obtaining an ingot of large grain size.

From each ingot thus prepared, two discs, 11 in. diameter and 1 in. thick, were machined, 11 in. being the largest dimension practicable. One face of each disc was ground and polished to produce a metallographic finish and then etched to reveal grain boundaries. The grain boundaries of one of each pair were masked with an insulating varnish (Araldite) and the grain interiors of the other were masked with the same material to leave an exposed channel, about 1 mm, wide, which contained the grain boundary.

With pure zinc and Alloy No. 5/2, the grain size was fairly large (often 1 in., rarely less than 1 in.) and the grain boundaries reasonably straight, so the masking operation could be done with a fair degree of accuracy. The grain size of Alloys Nos. 5/3 and 5/4, however, was more variable and the grain boundaries irregular in shape; the masking of the required areas, therefore, presented some difficulty. The masking of the grain boundaries was completed with reasonable precision and. with the corresponding specimen, most of the surface was masked, leaving only a few grain boundaries (those that were reasonably straight and between crystals of appreciable size) exposed. A brass rod was then screwed into a tapped hole in the cylindrical surface of the specimen and the preparation was completed by coating the remaining surfaces of the discs and the surface of the rod with Araldite varnish. A pair of these electrodes is shown in Fig. 5.

The electrodes were annealed at 120° C. for 3 hours to bring them to equilibrium (and to harden the varnish), then, after cooling to room temperature, they were immersed in N/10 potassium chloride solution saturated with oxygen, corresponding electrodes facing each other and stationed about 1 in. apart. The potentials of the electrodes were measured on open circuit at frequent intervals over a period of several hours against a saturated calomel electrode, but during the remainder of the test period corresponding electrodes were, of course, in closed circuit. The current flowing between corresponding electrodes was also measured frequently.

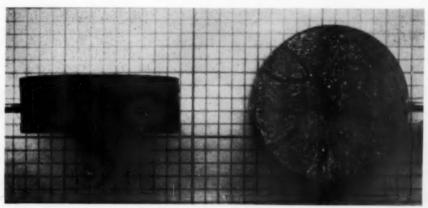
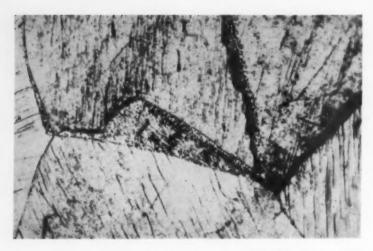


Fig. 5.—A pair of electrodes after tests had been completed. ×11 approx.



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Fig. 6.—Structure observed in quenched alloy after ageing for 72 hours at room temperature. Etched. ×1500

Current and potential had reached a fairly constant level in all cases after about three hours and the readings obtained at this stage are shown in Table V. The electrodes were left in position for a further 12 hours and readings were again taken, these also being shown in Table V. Inspection of the figures in this table shows that the current flowing is approximately proportional to the potential difference between corresponding pairs of electrodes, which indicates a cell resistance of the same order of magnitude in all cases. It may reasonably be concluded, therefore, that no great complications are

being introduced by polarisation.

With regard to the interpretation of the results, the fact that appears to be most significant is that when 0.10% aluminium is added to zinc, the potential of the grain boundary is moved 0.044 V. in the negative direction but that of the grain interior only 0.005 V. When interpreting potential changes at grain boundaries and grain interiors in aluminium-copper alloys, Mears and Brown concluded that the movement of grain boundary potentials in the negative direction was due to a decrease in the concentration of copper in solid solution in these zones, since copper in solution moves the potential of the alloy in the cathodic direction with respect to aluminium. Assuming that aluminium in dilute solution in zinc (when conditions are such that an adhering oxide film cannot form) exerts a potential of -1.7 V., and applying the same argument, these alterations in potentials may be taken to indicate that the aluminium in solid solution in the alloy is at a higher concentration at the grain boundary zone than within the grain.

This interpretation appears to be reasonable and in accord with established knowledge, but other interpretations are possible. For example, it is possible that, due to greater mobility, a larger number of aluminium atoms report at the reaction surface in the grain boundary zone, thus exerting an effect on the potential of these zones out of proportion to the actual aluminium concentration. An attempt was therefore made to examine the mobility of the aluminium in the zones of

the crystals of the alloys, and this experimental work is reported in the next section.

#### V. Diffusion at Grain Boundaries

The difficulties of determining whether or not the aluminium atoms in the grain boundary zone of an unsaturated alloy are more mobile than those within the crystal appeared to be considerable, whereas in supersaturated alloys the point can be investigated by comparatively simple means. An investigation was therefore carried out with a supersaturated alloy since it appeared that, from the data likely to be obtained, the conditions existing in a single phase (unsaturated) alloy might be assessed.

Cast bars of an alloy of composition C.S. Zinc + 0.65% aluminium were homogenised at a temperature of 350° C. and then quenched. A section was prepared as far as the "one nought" stage, re-homogenised at 350° C. for several days and

again quenched. The surface was prepared for metallographic examination, etched and examined at frequent intervals over a period of several months, the first examination being within five hours of quenching. The section was maintained at room temperature throughout the period and was re-polished and re-etched before each examination. In this way the course of the precipitation process was observed over a considerable period. The observations made may be summarised thus:

- A precipitate was visible at the grain boundary zones but not within the grains 24 hours after quenching.
- (2) When precipitation had become more general, it could be seen that in much of the boundary zone, but not in all of it, the particles of precipitate were substantially larger than those occurring within the grain.

These observations are illustrated by Figs. 6 and 7. The former shows the structure observed 72 hours after quenching; the precipitate at the grain boundaries can be seen quite clearly while the interior of the grain appears to be substantially free from precipitate. Fig. 7 was taken when the alloy had been aged for 21 days. Precipitation can now be seen to have occurred within the grain also, but in these areas the precipitate appears to be in a finely divided state, whereas in the grain boundary zones it occurs as appreciably larger particles that are more widely dispersed.

The mechanism of the process of precipitation from a supersaturated alloy is probably complex and is, at present, usually explained in terms of the dislocation theory. However, assuming that throughout the volume of a crystal the solute atoms are distributed at random (which is probably the case except when the solubility limit is approached), it may be concluded that if in one zone the particles of precipitate are fewer in number and larger in size, or appear sooner than those in a second zone, then diffusion must occur more rapidly

or more easily in the first zone. The evidence obtained from these experiments appears to justify the conclusion that in the grain boundary zones of a supersaturated zinc-aluminium alloy, diffusion does in fact occur more rapidly. The increased mobility of atoms in such zones is probably due to the peculiarities of the crystal lattice, the conjunction of two lattices of different orientation resulting in a zone of lattice misfit where "holes" and defects occur frequently\*. The greater mobility of the aluminium shown in supersaturated solutions is, therefore, due mainly to the properties of the zinc lattice and, since these properties are largely independent of the concentration of the solute, it is justifiable to conclude that in unsaturated solutions of aluminium in zinc. the aluminium atoms are more mobile in the grain boundary zone.

#### VI. The Effects of Impurities

The effects of a number of impurities on the incidence of intercrystalline corrosion in zinc-aluminium alloys were discussed in an earlier paper<sup>1</sup> (to which reference has already been made) and an attempt was made to evolve an explanation of the function of these impurities or additional alloying elements in either promoting or inhibiting the attack. However, no satisfactory explanation was found and one of the objects of the present programme of investigation was to examine this aspect of the subject in greater detail.

A number of alloys containing impurities or additional alloying elements were included in the experiments described in Sections II and IV and, in further persuance of the problem, the effects of the elements silver, gallium, thallium and indium when present in the alloys were examined, the experimental details and the results obtained being set out below.

#### Experimental Work

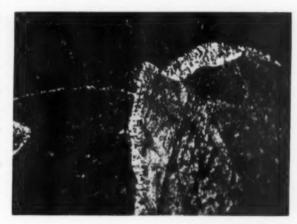
Alloys of the composition shown in Table VI were prepared and east into bars of  $\frac{3}{8}$  in. sq. cross section, the zinc used being of C.S. quality. These particular aluminium and magnesium contents were selected because they corresponded with the concentrations used

See Smoluchowski, for example<sup>10</sup>.

TABLE VI.—BFFECT OF IMPURITIES ON INCIDENCE OF INTERCRYSTALLINE ATTACK.

Basic Composition of Alloy: C.8. Zinc+4.0% Al+0.05% Mg)

Alloy No.	Impurity Added and Concentration	Type of Intercrystalline Attack and Depth of Penetration
6/1	0-002% Ag	Fairly frequent patches to a depth of 0-003 in.
6/2	0-010% Ag	Fairly frequent patches to a depth of 0-004 in.
6/3	0-020% Ag	Rather more frequent patches to a depth of 0.004 in.
6/4	0-025% Ag	As Alloy 6/2
6/5	0-001% Gn	Fairly frequent patches to a depth of 0.003 in.
6/6	0-010% Ga	More frequent patches to a depth of 0-003 in.
6/7	0-025% Ga	As Alloy 6/6 but to a depth of 0.004 in.
6/8	0-001% Tl	Fairly frequent patches to a depth of 0-003 in.
6/9	0-002° T	Fairly frequent patches to a depth of 0.004 in.
6/10	0-004% TI	Patches of more intense corrosion to a depth of 0.004 in.
6/11	0-008% Tl	As Alloy 6/10 but intensity of attack increased.
6/13	0-014% TI	Continuous layer to a depth of 0-007 in. Intense attack
6/13	0.025% TI	As Alloy 6/12 but to a depth of 0.020 in.
6/14	0-00025% In	Occasional patches to a depth of 0.003 in.
6/15	0-0005% In	Frequent patches to a depth of 0.003 in.
6/16	0.001% In	Fairly intense attack to a depth of 0.006 in.
6/17	0-004% In	More intense attack to a depth of 0.025 in.
6/18	0-008% In	Very intense attack to a depth of 0-025 in.
6/19	0.025% In	Very intense attack to a depth of 0.025 in.
6/20	Alloy of Composition	
	C.S. Zinc+4-0% Al +0-010% In	Fairly intense attack to a depth of 0.060 in.



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Fig. 7.—Structure of the same alloy after ageing at room temperature for 21 days. Etched. ×1500

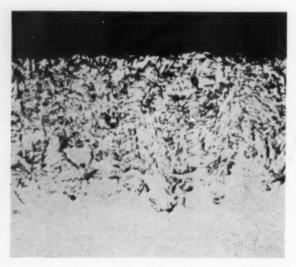
in the commercial alloy, the magnesium being added as a corrosion inhibitor.

Bars of each alloy were exposed to the action of an air/water-vapour atmosphere at a temperature of 95° C. for ten days in the conventional steam tank and the extent of the resulting intercrystalline attack was determined by metallographic examination. The results obtained are also shown in Table VI and the type of corrosion observed in the alloys containing thallium and indium is illustrated in Figs. 8 and 9.

It may be added, for the purpose of comparison, that commercial zinc-aluminium alloys used for pressure die casting, which are manufactured from high purity metal to Specification B.S.1004, are corroded only superficially when exposed in the conventional air/water atmosphere; a typical observation would be "occasional patches of intercrystalline corrosion to a maximum depth of 0.003 in."

The conclusions that emerge from this section of the investigation may be summarised thus:—

- The presence of silver and gallium in concentrations of up to 0·025% neither increases nor decreases the susceptibility of zinc-aluminium alloys to intercrystalline attack.
- (2) Thallium, at a concentration of 0·014% or more, increases greatly the susceptibility of alloys (of the composition indicated) to intercrystalline attack, the element being appreciably more noxious in this respect than either lead, tin, cadmium or bismuth.
- (3) The presence of indium in zinc-aluminium alloys (free from magnesium) increases to a considerable degree the susceptibility of these alloys to intercrystalline corrosion.
- (4) When small quantities of magnesium are also present in such alloys the efficacy of indium in accelerating the intercrystalline attack is most marked, the effect being quite noticeable at an indium content of the order of 0·001-0·002%, and very considerable at 0·004%. The intensity and depth of penetration of the attack increases with increasing indium content.



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Fig. 8.—Intercrystalline attack in alloy of basic composition; zinc with 4.0% aluminium and 0.04% magnesium, but containing, in addition, 0.025% thallium. Unetched.

#### VII. Discussion of Results

#### A. High Purity Zinc-Aluminium Alloys

The results obtained from the experimental work on zinc-aluminium alloys to which impurities or alloying elements were not added appears to justify acceptance of the hypothesis that the intercrystalline corrosion that occurs in single phase alloys is brought about by the selective oxidation of aluminium, and that the attack occurs preferentially at grain boundaries because the aluminium is at a higher concentration in these zones and may be able to diffuse more rapidly there than

within the grains.

In dry steam the corrosion is probably brought about by direct chemical reaction between water vapour and metal, but, since the rate of attack is greatly increased when liquid water is present, it appears very probable that under these conditions electrochemical action also occurs. Since aluminium is strongly anodic to zinc, an aluminium-rich zone in such alloys is likely to be anodic to a zone of lower aluminium content. The hypothesis put forward to explain the fundamental cause of the corrosion implies, therefore, that the grain boundary zones are anodic to grain interiors and, in pure alloys, electrochemical action may be visualised with the comparatively small grain boundary zone as anode and the larger grain interior as cathode. Experience has shown that such a combination is likely to bring about rapid intercrystalline attack. However, the electro-chemical experiments did not show the grain boundary zone to be anodic to grain interiors but, since the potential of the grain boundaries of zinc (originally 0.050 V. cathodic to grain interiors) was moved 0.044 V. in the anodic direction by the addition of 0.10% aluminium whilst that of the grain interior was moved only about 0.005 V., the existence of a powerful anodic component in the grain boundary zone is strongly

The presence or absence of a protecting film of corrosion

product may well be a critical factor in determining whether or not corrosion will occur at a particular zone in zinc-aluminium alloys. Both zinc and aluminium rely on the presence of a superficial film of corrosion product to stifle general attack, but if these films are disrupted at a given point corrosion will proceed in such an area. It seems possible that the lattice distortion associated with grain boundaries may decrease the adhesion between such films and the underlying metal in these zones, and if the presence of foreign atoms widens the zone of distortion the film may break down completely and facilitate local attack.

Since intercrystalling attack occurs when the alloys are exposed to dry steam at a temperature of 150° C., but not when heated in air at a similar temperature, it is likely that the film formed in air affords a greater degree of protection to the underlying metal. Winterbottom<sup>11</sup> is reported to have suggested that the presence of water vapour may alter the texture of the film of oxide formed on the surface of zinc at low temperature, rendering it amorphous in one case and crystalline in the other, but the relative protective properties of the two types of film are not known.

#### B. Alloys Containing Impurities and Additional Alloying

The experimental work described in this paper and in the earlier paper<sup>1</sup>, and by other investigators <sup>12–20</sup> has established the following relevant facts.

- The susceptibility of zinc-aluminium alloys to intercrystalline attack is decreased by the presence in the alloy of magnesium, copper, nickel and calcium, magnesium being most effective in this respect.
- (2) Susceptibility to attack is increased markedly by the presence of lead, tin, cadmium, bismuth, thallium and indium, and slightly by the presence of mercury and sodium.
- (3) Silver, gallium and iron have no noticeable effect on susceptibility to intercrystalline attack.
- (4) No intercrystalline corrosion occurs when magnesium, lead, tin, cadmium or gallium is present alone in pure zinc, but when magnesium and either lead, tin or cadmium are present together intense intercrystalline attack occurs.
- (5) The presence of magnesium in zinc-aluminium alloys counteracts the deletrious effect of lead, tin, cadmium, bismuth, thallium and indium when the concentration of these impurities is below what appears to be a limit for each element. At concentrations above this limit magnesium appears to be comparatively ineffective in this respect.
- (6) In dry steam, magnesium inhibits and lead accelerates the intercrystalline attack, while copper is comparatively ineffective.
- (7) When magnesium and copper are present in zincaluminium alloys, both the grain boundaries and grain interiors are rendered more cathodic than those of a pure zinc-aluminium alloy.

Accepting the explanation of the susceptibility of pure zinc-aluminium alloys to intercrystalline attack put forward in sub-section A above, it can be assumed that the attack will be accelerated if (1) the concentration and/or mobility of the aluminium in the grain boundary zone is increased; (2) a phase cathodic to the grain boundary

zone is created; (3) the grain boundary zone is rendered more anodic; or (4) the corrosion product at the grain boundary zone is rendered less adherent. If, however, postulates (1) to (4) occur in the reverse sense the attack will be retarded.

The corrosion accelerating elements, lead, tin, cadmium, bismuth, thallium, indium, mercury and sodium (a) have large atomic radii compared with zinc; (b) are almost insoluble in zinc (exceptions—cadmium and, to a lesser extent, mercury); (c) are cathodic to zinc and aluminium (exception—sodium), and (d) form no intermetallic compounds with either zinc or aluminium

(exception-sodium, which forms NaZn<sub>12</sub>).

It is established that lead, tin and bismuth, when present in zinc-aluminium alloys in excess of solubility limits, segregate at grain boundaries and, because of their low solubility and large atomic radii, thallium and indium may reasonably be assumed to segregate in a similar manner. When, therefore, any of these five elements are present, in excess of the solubility limit, in zinc-aluminium alloys, a cathodic phase is brought into being adjacent to the grain boundary zone and, due to the marked difference in atomic radii, the zone of lattice distortion is probably increased in width.

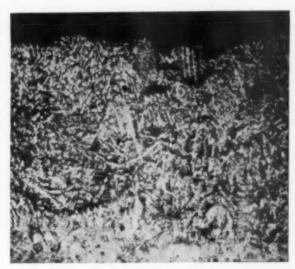
In conditions where a film of condensed water exists, electrochemical action will occur, the segregated impurities acting as cathode (in addition to the cathodic areas of the grains) and thus increasing the attack on the anodic grain boundary zones. Cadmium was shown to be considerably less effective in accelerating the attack than either lead or tin, while Lohberg reported that the effect of mercury was not marked. This is in accord with facts relevant to solubilities, since these elements would be dispersed throughout the crystal and not concentrated

at the corroding surface.

The action of the corrosion inhibiting elements does not appear to allow of a complete explanation in terms of simple electrochemistry. Copper, for example, is comparable in certain respects to cadmium in that it is cathodic to both zine and aluminium and exerts its effect when in solid solution, the effect, however, being opposite to that of cadmium. The electrochemical measurements showed that the addition of 0.30% copper to a single phase zinc-aluminium alloy moves the potential of both grain boundaries and grain interiors in the cathodic direction and reduces the current to very small dimensions. This appears to suggest that equilibrium segregation of copper is also occuring, which counteracts the effect brought about by the equilibrium segregation of aluminium. An objection to this explanation, however, is that if the copper were, in fact, present at the grain boundaries at a higher concentration it would be re-deposited as sponge during the corrosion process, thus creating a strongly cathodic component near the grain boundary, the effect of which would be to accelerate the attack.

The above considerations also apply to nickel, iron, silver and gallium, since these elements are also cathodic to zinc and aluminium and either inhibit, or have no effect on, the intercrystalline attack.

Of the corrosion inhibiting elements, magnesium is the most effective and this appears to be in accordance with theory, since the metal is anodic to both zinc and aluminium and may be expected to protect the more cathodic elements from attack. The electrode potential measurements made after 15 hours' immersion certainly



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Fig. 9.—Intercrystalline attack in alloy of similar basic composition but containing  $0\cdot025^\circ_{~0}$  indium instead of thallium. Unetched.  $\times\,100$ 

indicate that the grain boundary zones of the magnesium-containing alloy are less anodic than those of the magnesium-free alloy and this may be due, to some extent, to the fact that magnesium reduces the grain size of such alloys. Because of the much greater length of grain boundary associated with smaller grain size, the degree of equilibrium segregation occurring in such alloys is, presumably, less marked, and any cathodic impurities present are distributed over a greater area with a corresponding reduction in their effective concentration.

Whilst the effects of impurities in accelerating or inhibiting the intercrystalline attack in wet steam can be explained, to some extent, in terms of electrochemistry, the effects of these elements in dry steam, where electrochemical reaction cannot occur (see Table IV) must also

be explained.

The corrosion accelerating effect of lead (and other elements of large atomic radius) may be due to the increase in lattice distortion at the grain boundary zones brought about by the presence in these zones of large atoms, the rate of diffusion of aluminium in such zones, and thus the rate of oxidation, being increased. Alternatively, this increased degree of lattice distortion at grain boundaries may decrease the adhesion between the film of corrosion product and metal surface in such

areas, thus facilitating selective attack.

With regard to the corrosion-inhibiting elements, an observation that appears to merit consideration is that all such elements form inter-metallic compounds with aluminium, while none of the corrosion-accelerating elements do so. Recent work by Hill and Axon<sup>21</sup> appears to indicate that when two elements which form intermetallic compounds are in solid solution in a third, some degree of attraction exists between the atoms of the two solute elements. If this is a universal effect (not confined to magnesium and silicon in zinc) it is likely that if magnesium can diffuse less readily in zinc than can aluminium, the attractive force between the atoms may

reduce the mobility of the aluminium and thus reduce the rate of corrosion. Some measure of support to the assumption that the simultaneous presence of magnesium may decrease the rate of diffusion of aluminium is given by a statement by Evans<sup>22</sup> to the effect that the presence of magnesium in zinc-aluminium alloys delays the decomposition of the  $\beta$  phase by eutectoid transformation

The ineffectiveness of magnesium in counteracting the deleterious action of such impurities as lead and indium when these elements are present in concentrations above the critical limit (which is very low in the case of indium) appears to be due to the fact that these impurities form with magnesium intermetallic compounds which are decomposed rapidly by water vapour. Experiments reported earlier1 showed that the compound Mg2Pb is rapidly attacked by water vapour at room temperature and later work showed that the intermetallic compound MgZn<sub>5</sub>, when containing a small quantity of indium, is rapidly corroded by boiling water. This effect is, of course, additional to the cathodic action of these impurities in "wet" conditions.

#### Conclusion

The final conclusion that emerges from the investigation on the incidence of intercrystalline corrosion in cast zinc-aluminium alloys, part of which is described in this paper, is that the susceptibility of these alloys to intercrystalline attack in the presence of moisture can, for practical purposes, be counteracted by the addition to the alloys of magnesium provided the concentrations of impurities present, particularly indium, thallium, lead. tin, cadmium and bismuth, are below closely defined limits. These limits are defined in the British Standard

Specification relevant to the commercial alloys and are adhered to rigidly by reputable manufacturers of the

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"A major purpose of the company is to provide liaison between European and American technology, Dr. Williams stated in announcing the launching of the "Tremendous progress is being made new enterprise. in research and technology on each side of the Atlantic. yet—at the businessman's level—there are inadequate mechanisms for communications and integration of technological developments. As a result, benefits that could accrue to companies both in the United States and Europe are delayed in coming. What we are doing is to hasten the process of across-the-Atlantic information dissemination as it relates to technology, to markets for products, and to finance and investments.'

Dr. Williams, who introduced the concept of contract research in Europe with his establishment of the labora-

tories of Battelle Institute approximately ten years ago, said that among other things the new firm is advising American companies where to place research projects. In the technical information field, the Clyde Williams Corporation is collecting engineering and technological data for its clients and provides special information relating to technical products, new production processes, equipment, company capabilities, patents, licence agreements and related matters. Its market istudies include analyses of the expected role of foreign companies in world markets, statistical investigations of existing and potential markets, sampling surveys of consumer purchasing habits and motivations, and studies of European common market problems. A major activity will be investment counselling and the collection of information needed for the management of investment portfolios. This service will apply mainly to investments in technically oriented businesses and will be available both to American and European firms.

#### Pipe and Tube Order

An agreement for an initial supply of nearly £400,000 worth of seamless steel oil well casing together with tubing and linepipe for Petroleo Brasileiro S.A. (Petrobras), the Brazilian State oil company, has been signed by Stewarts and Lloyds, Ltd. The materials, part of which are for the Petrobras oil fields in the Bahia region, are being supplied from the Stewarts and Lloyds works at Clydesdale, near Glasgow, and Corby, Northants.

# Metallurgical Education and the White Paper

By J. Dunning, B.Sc., A.I.M., A.I.Prod.E.

Principal, Cleveland Technical College, Redcar

During the four years that have elapsed since the author was appointed Principal of the new Cleveland Technical College at Redcar, on Tees-side, he has been disturbed by the trends in the relative quality of the entrants to Ordinary National Certificate courses in chemistry and metallurgy. Of the chemistry entrants, 75% had G.C.E. "O" level passes in four or more subjects, as compared with 30% of the metallurgy entrants. At the other end of the scale, only 20% of chemistry entrants had no "O" level passes, in contrast to the 64% in the case of metallurgy. In the light of these figures and the recent White Paper on technical education, the author comments on the metallurgical aspects of the problem in this article and presents details of a course\* suitable for a technician qualification.

A T the beginning of this year, the White Paper "Better Opportunities in Technical Education" was presented to Parliament by the Minister of Education. Many of his ten proposals are operating already in technical colleges throughout the country. Nevertheless the White Paper was opportune if it awakened in the country as a whole a realisation of the changing pattern of technical education and the need to meet the economic challenge of our age. What is to be our next step? We have had time to examine the points made by the Minister and it is obvious that a number of of his recommendations ought to apply to the training of young people in the metallurgical industry.

#### Comparison with Engineering

It has been said of the engineering field that the technical college has not kept pace with the development of the industry. If this is true, it is equally so of the metallurgical sphere. So far, apart from the lowlevel operatives course, the only route open for the young entrants has been the National Certificate type of course. The distressing hall-mark of this system lies in the increased failure rates. This, to some extent, can be appreciated when due regard is paid to the fact that there is not only a marked increase in the number of students staying on in the grammar school sixth forms (something over 60% of the 1948 figures), but also an increase in the proportion of science students seeking University entrance. It is also conceivable that immediately after the war students pursuing a National Certificate course were older and, therefore, more purposeful in their desire to achieve a qualification. In effect, the calibre of students coming forward for a metallurgical course is not as strong academically as that of their older brothers. Let us, therefore, examine the problem and compare the position with that in the engineering field, as set out in Table I.

There are two significant omissions on the metallurgical side.

- (1) The Ordinary National Diploma.
- (2) The Technician Course.

#### **Ordinary National Diploma**

At a time when the Minister is recommending a shift of emphasis to sandwich and block release courses, surely there is a case for the "Joint Committee" to exercise its powers in providing an Ordinary National Diploma of

TABLE I.—COURSES AVAILABLE TO STUDENTS OF ENGINEERING AND METALLURGY.

Engineering	Metallurgy	
Higher National Diploma Higher National Certificate Ordinary National Diploma Technician Mechanical Oraft Practice	Higher National Diploma Higher National Certificate No equivalent course Operatives' Course	

two years duration on a six months sandwich basis. This would give the not-so-academically strong student twice the time spent on a three year part-time day release National Certificate course. Such a scheme would have the advantage of sustained study and time for consolidation. If, because of Ministry thought on non-specialisation at this level, the Joint Committee for Metallurgy is not prepared "to go it alone," then why not unite with the chemists and physicists to make a common Ordinary National Diploma scheme in science—a scheme embracing the fundamentals of chemistry, physics and mathematics, but with an emphasis on the practical and the opportunity for industrial training.

#### **Technician Qualification**

If educationalists and industry are prepared to sponsor this type of course, then two groups of students are to be considered: (1) the grammar school students who cannot measure up to four "O" level subjects; and (2) the "A" stream boy from a modern school.

The course would be of four years duration and ultimately the students could pursue, at the age of sixteen, a joint course of part-time day or block release study after the modern school boy had passed through a one year full-time course at the technical college—a course designed in consultation with industry, a course for which industry and the College Advisory Committee could help in student selection. This could be achieved with success in much the same way as pre-engineering courses are operated in many colleges. Why continue to be hidebound by a National Certificate scheme that in many instances is "untouchable" and psychologically unsuitable?

Is it necessary to gain a Higher National Certificate to be a shift chemist analysing for carbon, silicon, phosphorus, sulphur and manganese? Is it necessary to hold such a certificate to work in a routine mechanical

Since this article was written the scheme has been accepted by the largest iron
and steel concern in the author's "catchment area," and students will receive
instruction on this course as from September, 1981.

testing laboratory or on pyrometric control? There will be many who will argue that the student will be a better employee for having gained his qualification—and this is probably true if the student is able to pass through to his Higher National Certificate in four or five years. If he cannot, then frustration will set in and he will be poorer for an uncompleted course. How much more sensible to introduce a new course—a course that will examine practically and qualitatively. The following scheme is put forward if only to engender in industry a realisation of the value of an alternative course and to invite comment.

The course, as outlined in Table II, is planned for part-time day release with attendance on one day and one evening per week for the first two years, and one day and two evenings per week in the third and fourth years. There would be no difficulty in re-arranging the timetable to operate as a block-release scheme. The first and second years could serve two useful purposes: (1) as a common course for metallurgists and chemists; and (2) as a selection course for transfer to the Ordinary National Certificate or the technician final stage.

#### Syllabus

The details of the topics to be dealt with in the various sections of the course are presented below.

#### SCIENCE I

#### Chemistry

Theory-Structure of matter: physical and chemical change; elements, compounds and mixtures; atoms and molecules; solid, liquid and gaseous states in terms of elementary kinetic theory. Use of symbols and equations to represent chemical change. Methods of separating mixtures: solution and solubility; solubility curves: crystallisation; filtration; distillation; sublimation. Air and oxygen: formation of oxides; combustion and heat of combustion: acids, bases and neutralisation; simple acidimetric-alkimetric titrations. Equivalent weight and normality. Water and hydrogen: formation of water; formation of hydrates; reactions with metals and "activity series" of metals; preparation and production of hydrogen from (1) acids (2) water. Oxidation and reduction as illustrated by synthesis of oxides and reduction of oxides by coal gas. Law of simple proportions and percentage composition of compounds; empirical formulae. Classification of elements as metals and non-metals; brief survey of preparation and/or production, properties and reactions of the following elements and their oxides to illustrate metallic and non-metallic characteristics: carbon, sulphur, nitrogen, calcium, iron and copper. (Treatment of carbon to include (a) fuel gases, (b) carbonates and hardness of water.)

Practical—Separation of mixtures into pure components; types of chemical change; combustion and rusting in air; purification of compounds by recrystallisation, distillation and sublimation; solubility of air in water; solubility curve for potassium chlorate; water of crystallisation in hydrates; hardness of water; law of simple proportions; equivalents of magnesium and copper; preparation and properties of oxygen, hydrogen, carbon dioxide, nitric oxide, nitrogen dioxide; volumetric analysis (to include simple acid-alkali titrations only).

#### Physics

Theory—Units of measurement: length, mass, time. Accurate measurement of length: vernier, micrometer. Energy and energy transformations; work and energy. Mechanical energy: force as a push/pull producing change in state of motion; momentum; mass and weight; moments; centre of gravity. Measurement of mass: chemical balance; measurement of weight; Hooke's law

TABLE IL-OUTLINE OF COURSE FOR TECHNICIAN QUALIFICATION.

Year	r Subject	
First	English Mathematics/Drawing I Science I: Theory Science I: Practical	1 21 2 4
Second	English Mathematics/Drawing II Science II: Theory Science II: Practical	2 1 2 2 4 1
Third	Mathematics III Physics III Analytical Chemistry I Metallurgy I	21 21 3
Fourth	Project Physics IV Analytical Chemistry II Metallungy II	2 4 2 4 3 3

and spring balance. Measurement of density: direct measurement; Archimedes' principle and flotation; relative density; hydrometers. Fluid pressure: manometer and diaphragm type gauges; atmospheric pressure, mercury and aneroid barometers : Boyle's law : Charles' law. Heat energy: temperature and temperature scale; thermometers; transmission of heat applied to furnaces and vacuum-jacketed vessels; units of heat. Electrical energy: conductors and insulators; nature of electrical charge; resistance and Ohm's law; units of resistance, current and potential difference. Magnetic effect of electrical current : simple properties of magnetic substances; magnetic fields; lines of force; electro-magnets and uses in bells; relays and motors. Chemical effect of current: simple cell; Leclanche cells, accumulators. Heating effect of current; furnaces. Units of electrical power and energy.

Practical—Determination of density and relative density of solids and liquids; verification of Hooke's law; principle of moments; centre of gravity; verification of Boyle's law; constant volume gas thermometer; preparation and calibration of liquid in glass thermometers; comparison of heat conductance by various materials; radiation from different surfaces; verification of Ohm's law; field of force for bar magnet; field of force for straight conductor; solenoid.

#### SCIENCE II

#### Chemistry

Theory-Chemical activity and atomic structure; atoms consisting of nuclei and electrons; electron transfer and electrovalent compounds; electron sharing and covalent compounds; Octet rule for valency. Electrolysis; Faraday's laws of electrolysis and the Faraday; principles of coulometric analysis; electrode potential and activity series; selective discharge and applications in (a) extracting and refining metals, and (b) analysis. Chemistry of sodium and compounds (to include industrial production of hydroxide, carbonate, cyanide); chemistry of chlorine and hydrochloric acid (to include preparation and properties of chlorides of metals and non-metals). Oxidation-reduction as an electron-transfer process; important analytical applications, potassium permanganate, potassium dichromate, iodine. Brief survey of preparation and/or production, properties and reactions of the following elements and their oxides: magnesium, aluminium, zinc, lead, phosphorus and silicon. Gay-Lussac's law of combining volumes; Avogadro's hypothesis and consequences; examples from gas analysis; determination of vapour density and molecular weight; atomic weight. Simple qualitative treatment of reaction velocity, equilibrium and Le Chatelier's principle. Practical-Preparation of salts; verification of Faraday's laws: determination of the molecular weight of carbon dioxide; preparation and reactions of hydrochloric acid and chlorides; nitric acid and nitrates. Qualitative analysis: preliminary tests, (action of heat; flame test; charcoal block-reduction; action of dilute and concentrated

sulphuric acid); reactions of, and scheme for mixtures containing four of the following ions: lead, copper, iron, aluminium, zine, calcium, magnesium, sodium and ammonium; carbonate, sulphide, sulphite, nitrite, chloride, bromide, iodine, nitrate and sulphate.

Physics

Theory-Mechanical energy; graphical resolution of forces; parallelogram of forces; simple machines and efficiency; friction. Mechanical equivalent of heat. Specific heat and heat capacity; change of state and latent heat; simple calorimetry; determination of calorific value of fuels and food. Vapour pressure and hygrometry. equivalent of heat : resistance series and parallel groupings : shunts and series resistance in measuring instruments. Measurement of e.m.f. and resistance; potentiometer; Wheatstone bridge circuit; Post Office box; variation of resistance with temperature; platinum resistance thermometer. Disappearing filament pyrometer. Capacitance and capacitors. Thermocouple and applications in pyrometers. Rectilinear propagation of light; shadows; reflection of sound and light at plane and curved surfaces; refraction; critical angle; refraction through prisms; thin lenses: principles and use of Abbe refractometer, camera, microscope and telescope.

Practical—parallelogram of forces; pulley systems; inclined plane; mechanical equivalent of heat; specific heat and latent heat determinations; calorific value of coal; comparison of vapour pressures; electrical equivalent of heat; measurement of e.m.f. and resistance; effect of temperature on resistance; platinum resistance thermometer; thermocouples and temperature measurement; position of images with plane and curved mirrors; focal length of convex lens; use of Abbe refractometer; disappearing filament pyrometer.

#### Physics III

Theory—Wave nature of light; diffraction grating, visible spectrum and colour; ultra-violet, visible and infra-red radiations; emission and absorption spectra; spectrometer; flame photometer and colorimeters; applications in analysis; photometers and photometry; polarised light. Electromagnetic induction; dynamo; transformer; induction coil; telephone; induction furnace. Simple harmonic motion; sine curve. Capillarity; surface tension; viscosity.

Practical—Spectrometer; photometry; colorimetric analysis; use of flame photometer in chemical analysis; simple harmonic motion from vibration of springs and simple pendulum; capillary rise and surface tension; viscosity; diffraction phenomena; use of polarimeter.

#### Physics IV

Theory—A.C. theory: waveform; frequency; standard waveform-vector representation; inductance and capacitance in A.C. circuits; resistance, impedance and phase displacement; power and power factor; single-phase transformer; hysteresis and eddy currents. Thermionic emission and simple applications; diode; triode; photoelectric effect. Cathode ray tube. X-rays and X-ray diffraction; simple crystallography.

Practical—A.C. circuits; hysteresis; simple circuits involving diodes and triodes; cathode ray tube and applications; microscopic examination of crystals; photo-electric cells.

#### MATHEMATICS I

British and metric systems of weights and measures. Fractions; decimal fractions; significant figures; decimal places. Percentages; percentage error. Ratio and proportion: (examples from laws of chemical combination, density, concentration of solution). Squares and square roots. Simple equations and simultaneous simple equations; build-up and transposition of symbolical equations

(to include areas of rectilinear figures, surface areas and volumes of simple solids); change in value of function for given change in variable; changing the subject of a formula (involving simple fractions). Graphs: position of a point; rectangular co-ordinates; representation of pairs of variables from experiment and equations; interpolation and reading graphs; straight line graph and simple variation. Geometry and drawing: division of straight line into equal and proportional parts; construction of scales; construction of perpendiculars and parallels to lines; the circle; division of circle; measurement of angles; construction of regular polygons; loci.

#### MATHEMATICS II

Indices: positive integral indices: law of indices: roots as indices; logarithms to base 10; use of logarithms (four rules); the slide rule. Solution of equations, (i) simple factorisation of quadratics, (ii) general case for  $ax^2 + bx +$ c = 0). Approximate formulae for squares, cubes, square roots and cube roots of a binomial with one term small. Slope and intercept of linear graph : determination of laws by reducing to linear form and testing straight line : area under graph by mid-ordinate rule; graphs of quadratic and cubic functions: elementary ideas on slope of a curve at a point: approximate arithmetical determination of slope. Sine, cosine and tangent for angles not greater than 90°; solution of right-angled triangles by scale drawing and by trigonometrical ratios. Similarity of triangles and applications in optics. Areas: enlargement and reduction of areas by radial projection; construction of triangle, square and rectangle equal in area to square, rectangle and polygon; isometric and oblique projection; free-hand sketching

#### MATHEMATICS III

Collection of statistical data; their adequacy and reliability; presentation of data; simple classification and tabulation; mean, variance and standard deviation by calculations from a set of observations; permutations and combinations; probability; sampling. Logarithmic equations (i.e. index unknown). Introduction to calculus; gradients, graphical differentiation, first and second order differentials; maximum and minimum values; differentiation of (i) sums and differences, (ii) products, (iii) quotients; differentiation of  $x^n$ , sin x and  $\cos x$ ; integration as the inverse process to differentiation. Circular measure of an angle; ratios of angles greater than right angles; ratios of compound angles; graphs of  $\sin x$ ,  $\cos x$  and  $\tan x$  (O < x <360).

#### ANALYTICAL CHEMISTRY I

Theory-Methods of sampling solids, liquids and gases, Reactions in solution; ionisation in solution; conductance of solutions and factors affecting it; strong and weak electrolytes; Kohlrausch's law of the independent migration of ions; the law of mass action and application to solutions of electrolytes. Acid-alkali titrations; strengths of acids and bases; common ion effect; ionic product of water; pH and its determination; titration curves and choice of indicator; conductometric and electrometric titrations. Redox titrations: redox potential; choice of internal indicator. Solubility product; applications in gravimetric analysis; washing precipitate; fractional precipitation; applications in qualitative analysis. Theory of classical group scheme of qualitative inorganic analysis; preparation of solution; separation into groups; separation tables for main groups; (ions in the scheme: Pb², Ag⁺, Hg², Hg², Hg², Hg², Cu², Cd², As³, Sb³, Sn³, Sn³, Sn⁴, Fe², Fe², Al², Cr³, Co², Ni², Mn², Zn², Ba², Sr², Ca², Mg², K, Na⁺, Nh², CO³, Ni², MC³, Sp³, SO², NO¸, CN¬, F¬, Cl¬, Br¬, I¬, NO¸, SO², PO⁴, SiO², AsO³, AsO³, AsO³, CrO4-. No interference between radicals at this stage). Practical-Measurement of conductance of a solution:

measurement of pH using (a) capillator; (b) pH meter; variation of pH during acid-alkali titrations; conductometric titration for acid-alkali; volumetric analysis, comprising advanced acid-alkali determinations; potassium permanganate, potassium dichromate and iodine titrations; gravimetric determination of sulphate, iron, phosphate, and silica; qualitative analysis of mixtures of above ions without complications due to interference between ions.

#### ANALYTICAL CHEMISTRY II

Theory-Volumetric analysis; theory of precipitation reactions; use of silver nitrate and potassium thiocyanate solutions: internal indicators: simple theory of colloids: Transition metals and complex adsorption indicators. ions: properties of complex ions; co-ordination number; importance of complex formation in analysis. Theory of the use of E.D.T.A. in volumetric analysis. Theory of the use of snot tests in qualitative analysis; selectivity and sensitivity of reactions; desirable properties of reagents for spot tests. Use of organic reagents in gravimetric determination of metals. Use of colorimetric methods in analysis; distribution law and extraction of coloured materials from aqueous solutions. Gas analysis; revision of the gas laws, Avogadro's hypothesis; Henry's law, absorption coefficient; equipment and technique. Qualitative analysis; separation of metals in same group; modification of group procedure for interfering acids.

Practical—Volumetric analysis; use of silver nitrate, potassium thiocyanate and E.D.T.A. solutions; investigation of complex ion formation using ion-exchange resins; gravimetric determination of aluminium, nickel, copper, manganese; colorimetric determination of copper and iron; analysis of flue gas.

#### METALLURGY I & II

#### Extraction Metallurgy

This will be a two years' course. Candidates will be required to show a reasonable knowledge of fuels, refractories and the fundamentals underlying the extraction and refining of the non-ferrous metals—magnesium, aluminium, zine, tin, lead and copper—and of iron and steel.

Fuels—Classification, uses and properties of solid, liquid and natural and prepared gaseous fuels. Heat transfer, regeneration and recuperation. Conduction, convection and radiation. Heat exchanges and heat losses. Calculations in connection with combustion, gasification and heat balance. Elementary principles of electric heating.

Refractories—Requirements of a refractory material. Classification and manufacture. Chemical and mineralogical composition and physical properties. Selection of a refractory for a particular metallurgical operation, including insulators. Testing of refractory materials.

Extraction—Ores and their location. Mineral dressing; crushing, classification and concentration methods; calcination; roasting and sintering; flow sheets. Smelting: fundamental principles in hydro-, pyro- and electrometallurgical extraction. Furnace products; handling of dust and fumes. Refining: principles of fire and electrolytic refining; retreatment and recovery of by-products. Secondary metals: recovery and purification.

#### Physical Metallurgy

Solidification of metals and alloys. Lattice structure; crystalline nature and properties of metals and alloys. The metallurgical microscope—theoretical and practical considerations in the preparation and examination of metallurgical specimens. Equilibrium in binary systems: construction of equilibrium diagrams, thermal analysis, dilatometry, microscopic methods and X-ray methods. Effects of non-equilibrium conditions. Representation of simple ternary diagrams. Principles of heat treatment: diffusion, recrystallisation and grain growth; precipitation; phase transformations. Plastic deformation. Destructive and

non-destructive testing. Constitution, structure and properties of principal non-ferrous alloys. The iron-carbon equilibrium diagram. Corrosion: protection against corrosion; anti-corrosion alloys.

#### PROJECT

The content of this syllabus will depend largely upon the type of industry in which the student is employed. It is desirable that there should be close liaison with industry to ensure that the content of the course is closely linked with an evaluation of the processes and practices of the firm in question, and that the practical work will be translated into a thesis covering the whole of the year's work.

Throughout the four years, emphasis will be placed on the practical side of all subjects.

#### Conclusion

These suggestions are the outcome of a local condition. It is conceivable that other parts of the country are experiencing the same problems. If this is so, the advent of the White Paper provides the stimulus for new thought and development. Let us not miss the opportunity while it lies in our grasp.

#### AEI-Birlec Order from Czechoslovakia

As the result of visits by Mr. J. H. Crossley (export sales director) to Czechoslovakia, AEI-Birlec, Ltd., has received a contract worth £405,000. The contract calls for the supply of continuous tray pusher furnaces, gas carburising furnaces and sealed quench furnaces. Also included in the order are Birlec nitrogen generators. These furnace installations are all to be employed in the motor vehicle and tractor industry.

Two continuous tray pusher furnace installations will each comprise two linked gas-fired furnaces—one high and one low temperature—which will be served by a flow of atmosphere from two nitrogen generators. When installed, this plant will be used in the production of annealed pearlitic blackheart malleable castings. A continuous tray pusher gas carburising installation will comprise two gas-fired, single track, continuous furnaces, one of which will incorporate a hot and the other a cool oil quench. This installation will also have atmosphere provided by Birlec generators. This unit is for the production of gears for the motor vehicle industry. A further gas carburising furnace and two sealed quench furnaces will be utilised in the production of small components.

#### More Oxygen for Scunthorpe

SCUNTHORPE is to have a further 200 tons of oxygen a day for steelmaking. The British Oxygen Co., Ltd., are to add a new plant at their Warren-lane works to increase capacity to 600 tons a day. The new unit is scheduled to enter service towards the end of 1962, and. with additional liquid oxygen storage capacity, will cost nearly £750,000. A 200-ton plant is already piping gaseous oxygen to local steelworks from Warren-lane, and a similar unit is due to start operating late this summer. Most of the oxygen from the new tonnage plant will be taken by Appleby-Frodingham Steel Co. (a branch of the United Steel Cos., Ltd.) to feed their Ajax steelmaking furnaces. The remainder will be available to augment supplies for other oxygen processes at Richard Thomas & Baldwins, Ltd., Redbourn, and Lysaght's Scunthorpe Works (branch of G.K.N. Steel Co., Ltd.).

# Low Pressure Metallurgy

#### The Reduction of Oxides by Carbon

By A. S. Darling, B.Sc.(Eng.), Ph.D., A.M.I.Mech.E.

This is the third article by Dr. Darling under the general heading "Low Pressure Metallurgy" and the reference numbers follow on those in the earlier articles, the first of which took the form of a general survey, the second dealing with vacuum as an inert atmosphere. In discussing the reduction of oxides by carbon at low pressure, the author considered in the first instalment, published last month, the factors affecting the progress of reduction, including metal-crucible reactions, before dealing here with specific examples in the case of copper, molybdenum, chromium, ferrochromium and niobium.

(Continued from page 13 of the July issue).

#### TYPICAL EXAMPLES

#### Copper

Pure copper can dissolve only 0.0001% of carbon at 1,200° C. and 0.003% at 1,400° C. Because of this low solubility carbon is the ideal deoxidant for high conductivity copper. O.F.H.C. copper is generally produced by deoxidising molten cathodes with carbon, and casting in an atmosphere of carbon monoxide. By such techniques, oxygen contents of the order of 0.005% are attainable. Some of the unsoundness detectable in O.F.H.C. copper can be attributed to the reaction during solidification between dissolved carbon and oxygen, leading to effects similar to those caused by hydrogen and dissolved copper oxide.

The residual oxygen content of carbon-containing copper can be displaced to very low levels by reducing the partial pressure of carbon monoxide over the melt. For this reason, copper of very high electrical conductivity is readily attained by vacuum melting in contact with carbon. The specific gravity of such material is appreciably higher than that of copper melted at higher pressures.

Fig. 9, taken from data published by Stauffer, Fox and Di Pietro, 170 illustrates the effect of melting time at a pressure of 20 microns upon the density of 40 lb. charges of copper. The density curve approaches a value of 8.9438 after six hours of melting in a graphite boat. The oxygen content of copper melted at low pressure is considerably affected by the degree of vacuum maintained and, to a lesser extent, by the length of the melting cycle. Table III, which refers to 100 lb. charges, induction melted in graphite crucibles, emphasises the importance of low pouring pressures. This data illustrates that the advantages of long degassing periods are marginal compared with the reduced oxygen contents achieved by lower pouring pressures. This pronounced pressure dependence probably results from the low solubility of carbon in the melt. The equilibrium carbon



Degassing Time (hr.)	Pouring Pressure (micron)	Oxygen Content (wt.% × 10 <sup>5</sup> )	
1	38	8-8	
1	60	15-5	
3	24	8-1	
3	79	13-0	
0	14	4-6	
8	94	8-5	

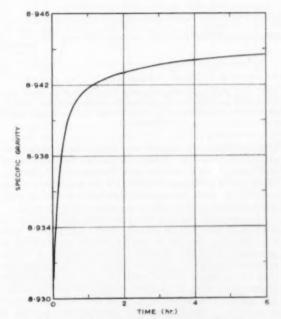


Fig. 9.—Density of vacuum melted copper as a function of time of melting. (Stauffer, Fox and Di Pietro<sup>170</sup>).

monoxide pressure will vary as the product of carbon and oxygen contents. To keep both these values low, a low pouring pressure becomes of primary importance.

Because shrinkage is not masked by gas porosity, pouring conditions have to be strictly controlled in order to obtain vacuum cast copper ingots free from a heavy central pipe. Low pouring rates are essential, approximately 100 lb. of metal per minute being a common value. Fig. 10 illustrates some pressure changes typical of those observed during the melting cycle of a 300 lb. induction copper melt finally poured at a pressure of 6 microns.

#### Molybdenum

The oxygen content of ductile powder metallurgy molybdenum is frequently of the order of 0.0025%. Arc melted molybdenum containing this quantity of oxygen is remarkably brittle. The low granular adhesion

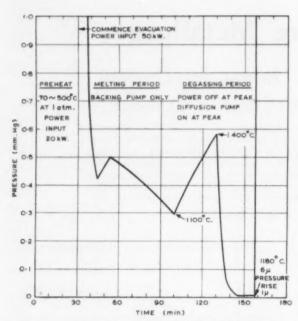


Fig. 10.—Pressure time curves during the vacuum deoxidation of a 300 lb. copper melt. (Stauffer, Fox, and Di Pietro. 170 Courtesy of Industrial and Engineering Chemistry).

of imperfectly deoxidised are melted molybdenum is attributable to the concentration of thin sheets of molybdenum oxide at the grain boundaries.<sup>171</sup> 0·01% of oxygen is sufficient to cover all the grain boundaries completely, while fractographic techniques disclose only discontinuous traces at the boundaries of ductile metal.

The favoured method of deoxidising molybdenum is with small additions of carbon. Parke and Ham171 were the first to report that by adding carbon and melting at pressures below 20 microns, excess oxygen could be removed and the reaction driven so close to completion that the residual carbon content was not appreciably higher than that of powder metallurgy molybdenum. The quantity of carbon required to deal with the oxygen in normal commercial quality molybdenum powder is commonly of the order of 0.01%. With the addition of carbon, oxides disappear and carbides become visible at the grain boundaries. The properties of the carbide are, however, different from those of the oxide. Arc cast molybdenum can be swaged with as much as 0.06% of carbon present. For this reason a slight excess of carbon over that theoretically needed to deal with all the oxygen present is not undesirable.

Microporosity is frequently observed in properly deoxidised molybdenum ingots.<sup>173</sup> This porosity is attributable to the small quantities of dissolved carbon monoxide which are thrown out of solution upon solidification. Such dissolved gas is not completely removed even when melting is carried out at pressures as low as 0·3 micron.

#### Chromium

Chromium has a strong affinity for oxygen, the free energy of formation of chromic oxide being approximately -90,000 cal./mole at the melting point of the

TABLE IV.—EFFECT OF OXYGEN UPON THE MELTING POINT OF CHROMIUM.

Investigators	Date	Oxygen Content	Melting Point (°C.)
G. Grube and R. Knabe (175) H. T. Greenaway	1936	~1.0	1,770-1,790
(176) N. J. Grant and	1951	0.02	1,835-1,855
D. 8. Bloom (177)	1952	Carbon deoxidised	1,885-1,915

metal. Molten chromium is capable, therefore, of extracting oxygen from any source having an oxidising potential higher than  $10^{-7}$  mm. of mercury. The partial pressure of oxygen in equilibrium with chromic oxide and chromium metal at  $1,000^{\circ}$  C. is  $10^{-24}$  mm. of mercury. Gulbransen and Andrew<sup>173</sup> confirmed that decarburised chromium oxidised rapidly on heating between  $600^{\circ}$  and  $816^{\circ}$  C., even at pressures below  $10^{-8}$  mm. of mercury.

The experimetal evidence suggests that chromic oxide is slightly soluble in molten chromium, and that a chromium-chromic oxide eutectic is formed.<sup>174</sup> Oxygen certainly depresses the melting point considerably, as the figures in Table IV indicate.

Considerable and determined efforts have been made to reduce the oxygen content of chromium in an endeayour to obtain material which is ductile at room temperature. Other body-centred cubic metals are very sensitive to traces of oxygen which diffuse interstitially into the lattice, and it is generally considered that chromium which is capable of being appreciably deformed at room temperature will have an inherently low oxygen content. It is doubtful, however, whether a critical assessment of the experimental data leads to the belief that oxygen is the main factor affecting the mechanical behaviour of chromium. Although, for example, oxygen affects adversely the high temperature ductility of chromium. it has little effect upon the brittle-ductile transition The limiting oxygen contents so far temperature. achieved have been of the order of 0.003%, and it is conceivable that very much lower values might result in improved room temperature ductility.

Although hydrogen can be employed to reduce the oxygen content of chromium the reaction is very slow, and the use of carbon under low pressure conditions is much to be preferred. Fig. 11, illustrates the manner in which carbon monoxide pressure affects the carbon-oxygen relationship in pure chromium at 2,000° C.178 At pressures of 5–10 mm. of mercury approximately  $0\cdot2\%$  by weight of carbon is required to reduce the oxygen content to  $0\cdot01\%$ . Excessive volatilisation of chromium occurs if the pressure is reduced much below 5 mm.  $^{178}$  At 2,000° C. the vapour pressure of chromium is 50 mm. of mercury, that of copper being only 35 mm. at the same temperature.

Some workers have, however, melted chromium and its alloys at pressures below 0·1 mm. of mercury without excessive volatilisation.\footnote{157} Metal-crucible reactions present the greatest difficulty. The severity of this effect is demonstrated by the fact that pure chromium, melted in an alumina crucible at 1,900° C. in vacuo, picks up 0·2-0·3% of aluminium. Thoria, which is not wetted by molten chromium is a better refractory. By using low melting pressures it becomes possible to drive the carbon-oxygen reaction so that both of these constituents are reduced to very low levels. Fig. 12 illustrates some pressure-time curves observed by Sully,

Brandes and Provan during the melting and deoxidation of a 90:10 chromium-cobalt alloy containing a slight excess of carbon. The first pressure peak is caused by hydrogen evolution from the electrolytic chromium. When the chromium begins to melt, it takes into solution some of the cobalt, thus initiating the carbon boil. The subsidiary pressure peaks reflect local melting and solidification during the heating period. A sudden drop in pressure occurs when the whole charge becomes molten. Oxygen contents as low as 0.06% are obtainable by suitable choice of casting pressure. If, however, the carbon content is reduced much below 0.02-0.03%, crucible reaction results in an increased oxygen content.

Due to the formation of stable carbides, chromium melts containing titanium and zirconium cannot be efficiently deoxidised by carbon at low pressures.

#### Ferrochromium

A high carbon content is detrimental to the performance of chromium alloys such as stainless steels and heating elements of the nickel-chromium group. Rohn, at Heraeus Vacuumschmelze, developed in the 1920-1930 period several processes for the decarburisation of such alloys by vacuum melting under conditions which permitted fairly rapid reaction between the dissolved carbon and chromic oxide. These processes were applied largely to nickel chromium production, and Kroll<sup>24</sup> reports that similar experiments with stainless steels met with little economic success.

Vacuum processes are now being successfully employed for the production of low carbon ferrochromium. The solid state reaction between carbon and chromic oxide proceeds rapidly in vacuum. Kroll and Schlechten^{179} demonstrated in 1948 that at 1,460° C. in a vacuum of 8 microns, carbon contents as low as  $0\cdot38\%$  could be achieved. One year later the Union Carbide Company announced a vacuum process for reducing the high carbon content of electric-furnace ferrochromium by using silica as an oxidising agent.  $^{189.\ 181}$ 

This reaction is also carried out in the solid state. Ferrochromium containing approximately 68% of

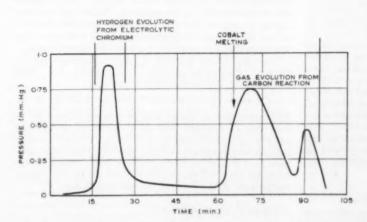


Fig. 12.—Pressure changes over a melt of 90: 10 chromium-cobalt deoxidised with carbon. (Courtesy of Institute of Metals).

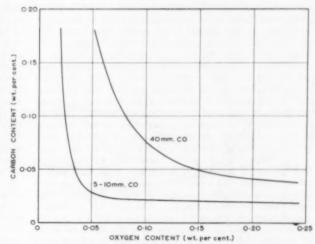


Fig. 11.—Effect of carbon monoxide pressure on the carbon and oxygen contents of molten chromium at 2,000°C. (J. H. Moore. 172 Courtesy of Vacuum Symposium Transactions, 1954).

chromium, 27% of iron, and 5% of carbon is ground to a particle size of 30 microns and mixed with the appropriate quantity of finely ground silica rock. The resultant mixture, bonded with chromic acid, is pelleted and charged into a retort subsequently evacuated to pressures below 2 mm. of mercury. At temperatures in the vicinity of  $1,250^{\circ}$  C. the reaction is very rapid and results in a product containing 0.01% of carbon and 6% of silicon. This high silicon content is beneficial, since it can be utilised to deoxidise the metal bath to which the pellets are finally added.

The vacuum oxidation process is carried out at temperatures high enough to maintain a satisfactory rate of reaction, but low enough to prevent melting of the pellets. The incipient fusion which tends to occur at the higher carbon contents forms a skin round the pellet surface, thereby reducing the reaction rate. Much of this tendency towards fusion can be overcome by partly

oxidising in air the pellets of high carbon ferrochromium before the vacuum oxidation process, <sup>182</sup> which is then carried out in the vicinity of the solidus. Fig. 13 illustrates diagrammatically the effect of carbon content upon the melting point of a 70% chromium alloy having a chromium: iron ratio of approximately  $2\cdot5:1$ . When the carbon content has been reduced below the eutectic point, temperatures can be increased above that of the solidus as the amount of liquid formed in this region is correspondingly low. The optimum heat treatment zone is shown on Fig. 13 as a shaded band.

#### Niobium

Niobium displays a great affinity for oxygen, which has a considerable effect upon its physical and mechanical properties. 0.9% of oxygen increases the hardness from approximately 90 to 340 D.P.N.,

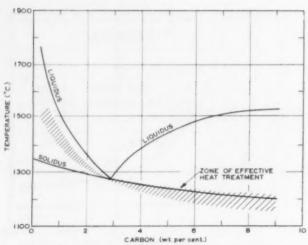
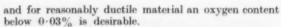


Fig. 13.—Effect of carbon on the melting point of ferrochromium alloys. Zone of effective decarbonisation is shaded.



At the present time ductile metal is largely produced by powder metallurgical techniques which permit compacts to be sintered in high vacuum well away from any oxide-containing refractories. Oxygen enters into solid solution with the metal to the extent of 1% by weight at 1,100° C. The free energy of formation of the lower oxide (NbO), is approximately 107,000 cal./mole of oxygen at 2,000° C. The dissociation pressure of this oxide is, therefore, of the order of 10–12 mm. of mercury, and the dilute oxygen solutions are even more stable.

Decomposition of the oxide does not occur to any extent at temperatures below the melting point of 2,468° C. At 2,000° C. the oxygen solubility is approximately 1·9% by weight. The increase in dissociation energy with decrease in dissolved oxygen has been estimated as being approximately 10 k. cal./mole O<sub>2</sub>/per power of ten in oxygen concentration. <sup>195</sup> The powder metallurgy process depends largely upon deoxidation with carbon at temperatures in the range 1,700°-1,800° C., after which the last traces of oxygen are removed by volatilisation between 2,100° C. and 2,300° C. <sup>187</sup>

The two-stage process is rapid and economical. If all the oxide were evaporated, metal losses would be of the order of 5%. By reducing the oxide, rather than evaporating it, weight losses are kept down to 1%. <sup>185</sup>

Efficient purification by the two-stage reduction method requires careful control of the carbon additions. The virgin powder commonly contains approximately 0.5% of oxygen and up to 0.3% of carbon. The optimum ratio appears to be that which ensures enough carbon to remove two thirds of the oxygen present as carbon monoxide. Intentional carbon additions may be required to achieve this ratio.

#### Mechanism of the Niobium Reduction Process.

Metallographic evidence indicates that the added and dissolved carbon reacts to form a carbide with the metal at an early stage of the reduction process. 185 At tem-

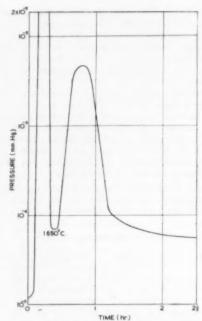


Fig. 14.—Pressure changes observed by Williams during the deoxidation of nioblum by carbon.<sup>107</sup> (Courtesy of the Institute of Metals).

peratures in the vicinity of  $1,700^{\circ}$  C., the reduction process can be represented as follows:—186

$$Nb_0O_5 + 5NbC \implies 7Nb + 5CO$$

Before combination can occur carbon and oxygen must diffuse independently to a free surface, Such diffusion, being interstitial, is likely to be rapid, and O'Driscoll suggests that the reaction rates are controlled primarily by the low rate of solution of carbon in niobium. Miller<sup>185</sup> remarks, however, that the sintering time for large bars becomes excessive, and recommends a preliminary sintering process followed by cold rolling and a final sinter. Williams<sup>187</sup> emphasises the importance of sintering under conditions which expose a large surface area so as to facilitate the reaction. These observations appear to suggest that the controlling factor is either the area of the exposed surface or the partial pressure of the carbon monoxide immediately above the exposed surface.

The equilibrium pressure of carbon monoxide above niobium containing oxygen and carbon has been estimated by Richardson as follows:—

$$P_{co}$$
 (mm. Hg.) = 3 × 10<sup>-2</sup> (Wt.% O) (Wt.% C)

This expression indicates that pressures of the order of  $10^{-5}$  mm. of mercury will be required to reduce the oxygen content of niobium to  $0\cdot03\%$ . Williams experimental results agree so closely with the thermodynamic data that it appears very probable that limiting conditions are achieved on, or immediately above, the surface rather than in the body of the reacting masses.

#### Niobium Production Processes

Niobium is successfully sintered in equipment having an ultimate pressure of  $1 \times 10^{-5}$  mm. of mercury.

Although oil diffusion pumps are satisfactory, efficient baffling is required. An essential feature of the process is the provision of a suitable mixture of raw and recirculated niobium powder. 187 The virgin powder has a true particle size distribution of 0.01-5 microns. Due to aggregation the effective particle size range is 1-50 microns. Bars sintered from virgin powder alone blister badly due to the development of high internal gas pressure. By employing a coarse grade of recirculated powder a high proportion of open porosity is retained during sintering, and the bars can be heated rapidly without any tendency to burst open. The recirculated powder is obtained from degassed hydrided niobium scrap. Use of this material facilitates the sintering and improves the economics of the process by

permitting the recovery of clean scrap. Acceptably low oxygen values necessitate effective pumping speeds at the surface of the bars of 3 litres/sec./ g. of material sintered. Under such conditions oxygen contents within the range 0.005-0.018% can be obtained after ten hours sintering. Fig. 14 illustrates a pressuretime graph obtained during the sintering of a 2 kg. bar in a system having an effective pumping speed of 6,500 litres/sec. 187 The initial rapid increase of pressure is due to the evolution of hydrogen. This evolution is complete at 1,650° C., and at higher temperatures the carbon reduction process commences. Approximately 40 minutes are required to raise the temperature of the bar from 400° to 2,300° C. Most of the shrinkage occurs between 1,600° and 2,000° C. The sintering time at maximum temperature varies from 2 to 10 hours. The final pressure, arrived at asymptotically, affords an effective demonstration of the accuracy of the estimated equilibrium pressure, and indicates that still lower pressures would permit the attainment of lower oxygen contents.

In view of recent developments in the field of electron bombardment it appears likely that this melting process will eventually displace powder metallurgy as a large scale production technique. Operating above the melting point avoids temperature limitations and realises all the the advantages associated with refining processes carried out in large baths of molten metal. Because a temperature increase of several hundred degrees is thermodynamically equivalent to a tenfold improvement in vacuum, melting by electron bombardment should facilitate the production of ductile niobium of low oxygen content.

#### Conclusions

Even the most refractory oxides can be quantitatively reduced by carbon in vacuum. If the object is to produce a completely oxygen- and carbon-free metal, however, the process is limited to those metals, nonvolatile at the reaction temperature, in which the tendency towards reduction is considerably greater than that leading towards carbide formation. Low pressure carbon reduction processes have been successfully employed for refining pure iron and its alloys, nickel, copper, molybdenum, chromium and niobium. There appears to be no reason why cobalt, vanadium, tantalum, tungsten and uranium should not be successfully treated by the same technique. Whether the reaction is regarded as an oxidation or a reduction depends upon the result desired. Because the reaction can be driven to completion in vacuo, carbon can be effectively eliminated, when desired, by controlled oxide additions.

Fundamental practical difficulties are associated with reduction or decarburisation processes which are carried out in the solid state. Many metals such as chromium are capable of dissolving or being dissolved in their own oxide. In the latter instance the first stage of the reduction process would involve the saturation of the oxide with the pure reduced metal. Only when this has occurred will discrete particles of the metal separate. In many instances these particles will be saturated with oxygen. Due to the agglomeration of such particles the mixture sinters and becomes denser. Evolved gases have difficulty in escaping, and the partial pressure of carbon monoxide in the reaction zone increases, thereby slowing up the reduction.

Such considerations make it essential to ensure that compacts or briquettes are pressed and sintered under such conditions that a large and effective surface area expedites the reaction. Maintenance of this large surface area will probably involve crushing and repressing, or mechanical working operations on the compact at various stages during the reaction. Close control is required to ensure that the reaction is pushed to completion.

Reductions in the liquid state are simpler to operate providing that working temperatures are not too high. Above 1,500° C. reaction between molten metals and refractory materials makes it difficult to ensure a completely oxygen-free melt.

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Fig. 1.—General view of the unloader showing the trolley over the hold of the ore carrier.

# New Electrically Operated Travelling Ore Unloader for Cargo Fleet Iron Works

ESIGNED and constructed by The Wellman Smith Owen Engineering Corporation, Ltd., a 13½ ton (gross capacity) electrically operated travelling ore unloader has recently been installed at the Cargo Fleet Iron Works of the South Durham Steel and Iron Co., Ltd. The general features of the machine can be seen from the illustrations. It is fitted with a water-side hinged boom giving clearance for ships when raised, and a fixed boom forming an extension of the traversing track on the land side. The trolley, which runs on the bottom boom of the main girder, is equipped with an underhung slewing type jib and carries the main machinery house and the driver's cab.

The unloader is carried on twenty-eight wheels, sixteen of them running on the water-side double rail tracks. The reason for this track layout is that the maximum permitted wheel load is 30/33 tons, in keeping with the carrying capacity of the wharf. The outreach of the hinged boom allows the grab to handle ore from 14,000 ton carriers having a 63 ft. beam, and a maximum lift of 80 ft. enables the grab to take ore from the bottom of the carrier hold under low water Spring tide conditions. The nett capacity of the ore grab is 6 tons, and when handling ore with a density of 1 cwt./cu. ft. an output of 400 tons/hr. can be achieved. The main dimensional details are as follows:—

Span (centre to centre of tracks)	46 ft.	10‡ in.
Rail centres (wharf side tracks)	12 ft.	21 in.
Maximum height of lift	80 ft.	0 in.
Ground rail level to bridge rail level	67 ft.	6 in.
Maximum reach of grab from wharf side	55 ft.	0 in.
Slewing radius	14 ft.	0 in

The power supply for the trolley, boom hoist and traversing hopper motors is 230–240 V. D.C. and that for the long travel, rail clamp and conveyor motors 440 V., 3 phase, 50 c./s. A.C. Details of the operating speeds and motors is given below:—

Trolley			
traverse	500 ft./min.;	2- 72 b.h.p. at 700 r.p.m.	
Grab hold	200 ft./min.;	1-100 b.h.p. at 550 r.p.m.	
Grab close	200 ft./min.;	1-100 b.h.p. at 550 r.p.m.	
Slewing	4 ft./min.;	1- 25 b.h.p. at 700 r.p.m.	
Boom hoist	12 ft./min.;	1- 25 b.h.p. at 700 r.p.m.	
Long travel	90 ft./min.;	4- 30 b.h.p. at 960 r.p.m.	
Spotting hop			
traverse	60 ft./min.;	2- 8 b.h.p. at 700 r.p.m.	
Rail clamps		2- 5 b.h.p. at 945 r.p.m.	
Boom latch		2 thrustors	
Conveyor		2- 15 b.h.p. at 720 r.p.m.	

#### **Loading Facilities**

During normal operation the grab transfers ore from the carrier to a large hopper placed on the centre line of the water-side leg structure and immediately over one end of a 3 ft. 6 in. wide conveyor belt. The ore passes from the hopper on to a drop-bar grizzly feeder from which it is transferred to the belt conveyor which runs at an inclined angle across the wharf to the land side of the machine. The conveyor is 80 ft. long and, together with allied equipment, is built into the structure on the unloader and moves with it.

On the landward side of the unloader, two ore transfer tracks at 22 ft. centres run along the length of the wharf on either side of a storage bunker. At the top end of the conveyor there are three loading valves which allow the ore to flow into wagons on either track or, alternatively, into the bunker.

Placed at a low level on the main structure are two motor-operated spotting hoppers which have a traversing distance to cover three lines of railway tracks. These hoppers are used for transferring ore from the grab direct into railway wagons, and come into use in the event of the conveyor equipment being out of action. Again at the extreme landward side of the unloader there is a fixed spotting hopper placed on the centre line of the outer transfer car track, and it is possible for the grab to feed ore direct into this hopper as a further alternative.

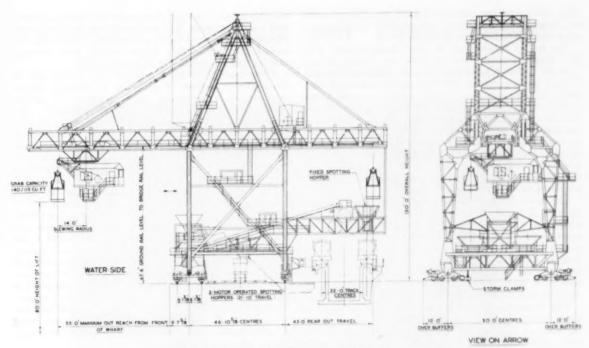


Fig. 2.—Side and end elevations of unloader.

#### Structural Design

The structure is built up of rolled steel sections and plates securely riveted together and adequately braced in all directions to stand the loads and stresses resulting from heavy operational duties. The bridge consists of two parallel steel girders of lattice construction, carried by two structural legs of the portal type designed to permit the trolley to pass through, and at the same time give clearance for the slewing jib. The legs are crossbraced in a direction at right angles to the track, a portal opening being formed under the bottom girders for the passage of railway trucks. Wherever possible broad flanged beams have been used in preference to built-up members, and the main members of the legs follow this construction. The trolley runway tracks extend the full length of the bridge and accommodate air-hydraulic buffers at each end.

When in the lowered position the weight of the hinged boom is taken by steel folding ties pivoted at the top of the tower, which is of apex construction securely braced to the top of the portal legs. Special attention has been given to the necessity for preserving rail alignment between the hinged and fixed runways, and heavy forged steel brackets of special design are fitted to the fixed track joists to act as a centring and aligning device when the cantilever boom is lowered into position. The two sets of tracks are maintained practically level and this, in turn, has a beneficial effect on the riding qualities of the trolley. The entire structure is well provided with stairways and platforms giving easy access to all mechanical parts for purposes of adjustment and repair.

#### Long Travel and Rail Clamps

The unloader is carried on compensated bogies

arranged under each corner of the main tower, the bogies being attached to articulated rocker beams which, in turn, are pivoted in the lower end of each leg of the tower. The arrangement of the long travel gear on the water-side is of a special nature, since the tracks are at 12 ft. 2\(\frac{1}{2}\) in. centres, which means that, while the construction is carried out as described above, the articulated rockers are connected by equalising girders to the structure.

Each bogic carries 2 ft. 6 in. diameter centre-flanged travelling wheels, the wheels being gun-metal-bushed and running on fixed axles. The travel motor at each corner of the structure is mounted on the rocker beams and drives the runners through suitable worm and spur gear reductions. Centre-flanged wheels are necessary because each track consists of double 109 lb./yd. F.B. rails. Automatically applied rail clamp gear is provided on all tracks.

Control of the four 30 b.h.p. long travel motors is simultaneous, and allows for plugging and primary resistance acceleration in both directions: each motor is provided with a solenoid brake. The travel and clamp control gear are interlocked to ensure that travelling can only take place when the rail clamps are released.

#### Trolley

The trolley frame and hanger are combined to form one rigid structure, the hanger terminating in a circular steel path to take the slewing guide rollers which are attached to the central mast. The mast is of plated construction, and fitted into it at the top is a heavy casting which carries the main pivot centre pin. The jib is of lattice braced construction, designed at the rear to

carry the contactor house and main machinery house enclosing the hoisting motors and gearing, the operator's

cabin being suspended underneath.

The trolley is mounted on four double-flanged 2 ft. 6 in. diameter runner wheels, both axles being driven, each by a 72 b.h.p. motor through double reductions of spur gearing. Both traverse motors are controlled simultaneously and allow of plugging and series resistance control in both directions, also incorporating the Wilson-Ritchie dynamic braking feature. The equipment is designed for use with slow-down in addition to final traverse limit switches, the former arranged to retard the speed of the trolley towards the ends of the track, and later to cut out the motors and apply the brakes immediately before the buffers are contacted. Built-in jacks are fitted at each corner of the trolley.

The slewing gear is also mounted on the trolley frame, and the 25 b.h.p. motor drives through worm and spur reductions to the main spur rim, which is fitted to the mast centre-pin casting. Mounted at the top of the centre pin is a rotating contact column with a sufficient number and size of rings to take the mains and supply the traverse and slewing motions. The slewing gear control is arranged for plain series in both directions, and this motion is supplied with an M-type brake.

The Priestman grab is of the four-rope heavy-duty type, having a flush capacity of 115 cu. ft. and a heaped capacity of 140 cu. ft. The grab jaws are fitted with detachable manganese steel teeth. The grab hoisting gear is of the double winch type suitable for speedy manipulation by the crane driver, and there are no clutches to be operated. The holding and closing barrels (3 ft. 6 in. diameter) are each driven by a 100 b.h.p. motor through worm gearing, each set of gearing being lubricated by an individual oil pump.

The design of the hold and close line equipment allows of series resistance control in the hoisting direction, with dynamic braking and potentiometer control lowering. In the closing line there is included a special jamming relay designed to re-insert resistance into the motor circuit in the event of the grab blades encountering some obstacle they cannot crush, or if the hold line motor has not accelerated before the grab is clear of the material. Both these motions are fitted with Igranic M-type brakes and hoist limit switches.

For maintenance purposes in the machinery house a 4 ton pulley block running on a centrally disposed track is provided, the height of lift of this block being such that any unit in the machinery house can be lowered to ground level when necessary. The trolley is protected against the weather by a substantial steel cover having a removable roof and arranged to facilitate inspection of

the interior.

#### Boom Hoist, Spotting Hopper and Conveyor

The boom hoisting gear is housed in a separate machinery house situated on the landward side of the apex frame and at the top level of the main girders. The gear, which is mounted on a separate machinery frame attached to the main structure, consists of two 4 ft. 3 in. diameter barrels driven by a 25 b.h.p. motor through the medium of spur gearing. The boom latch gear consists of two massive steel hooks, pivoted below the top of the apex structure, designed to secure the hinged boom in its raised position, thus relieving the lifting ropes of all load. These latches engage automati-

cally by gravity and are disengaged by means of thrustors. This motion is fitted with an M-type brake together with limit switches in both directions, while the control gear gives plain series hoisting and dynamic braking and potentiometer control lowering.

At the base of the main structure and on each side of the conveyor system is provided a spotting hopper mounted on a traversing trolley carried on girders running between the main legs of the unloader. The travelling distance of each hopper—21 ft. 10 in.—facilitates the loading of ore into wagons on any one of three tracks. Each trolley frame is mounted on four runners and rope hauled by a 8 b.h.p. motor. An Igranic M-type brake is fitted to each motion and control is plain series in both directions.

The Ross drop-bar grizzly feeder under the main receiving hopper has working centres and width of 7 ft. 9 in. and 2 ft. 6 in., respectively, the variable speed drive being provided by a 15 b.h.p. motor through worm and spur gear reductions. A similar drive is installed at the conveyor head pulley, but through chain and worm gear.

As previously mentioned, the flow of ore at the top of the conveyor can be directed to either of two transfer cars or into the storage bunker, this being made possible by the provision of three power-operated Ross wagonloading valves. The operation of the valves is by compressed air, the compressor and auxiliary plant being situated in the machinery house mid-way in the length of the conveyor system.

#### **Control Cabins**

The operator's cab, housing the controllers for all trolley motions, is situated in a most advantageous position below the main machinery house and on the centre line of the mast, giving the operator an unobstructed view of his load in all positions. Control of the boom hoist and long travel motions is carried out from the boom hoist machinery house and the long travel control cab on the tower structure, respectively.

#### **Electrical Equipment**

All motors on D.C. supply are of the totally enclosed one-hour rated series-wound crane type, manufactured by The General Electric Co., Ltd. On A.C. supply, the long travel motors are of the slip-ring type, and the rail clamp motors of the high torque squirrel-cage type, all motors being of weatherproof construction.

The control gear throughout is of Igranic manufacture, comprising enclosed contactor panels with master controllers for all motions except the rail clamp contactor gear, which has a pedal operated switch, and the spotting hopper traverse and conveyor motions, which

are drum-controlled.

The long travel collectors A.C. and D.C. are mounted on the landward side of the unloader structure, making contact with T-bar conductors which are carried from the adjacent transfer car track structure. Current is conveyed to the trolley by Brecknell Willis collectors making contact with overhead T-bars which run the length of the bridge. The entire collector system is fully protected from rain and snow.

The unloader is fitted with the usual wind pressure indicator gear, so that when the velocity on the anemometer exceeds a certain figure, a relay automatically cuts out the travelling circuit and prevents the storm

clamps being raised.

# Furnace Research and Development

New Premises for AEI-Birlec Laboratory



General view of the research and development department.

HE charge has been levelled against British furnace builders that they are not engaging in research to the extent that their critics feel they should, preferring rather to buy designs abroad. That this is by no means universally true was amply demonstrated recently when AEI-Birlec, Ltd., invited representatives of the press to see their new research and development department. It is true that AEI-Birlee has had-and still has-arrangements with both American and Continental furnace builders to manufacture to their designs in this country under licence, but it would be wrong to take that as evidence of the company's lack of appreciation of the importance of carrying out research and development work on its own behalf. On the contrary, a department for this purpose came into being nearly twenty-five years ago, soon after the formation of the company. For the greater part of its existence, it has been located at the main Birlec premises at Tyburn Road, Birmingham, but in recognition of the growing importance and scope of its work, the department was transferred last year to the present larger premises at Wood Lane—a short distance from Tyburn Road—adjacent to the works training school established some five years

The new laboratory is housed in a building 275 ft. long × 70 ft. wide, which, including a balcony, provides a floor area of 20,000 sq. ft. It is believed that this is the largest and best equipped research and development department in the furnace industry.

Electric power up to a rating of 750 kVA. is distributed throughout the department, as is town's gas up to 9,000 cu. ft./hr., mains water up to 4,000 gal./hr., recirculated cooling water up to 2,000 gal./hr., and compressed air up to 18,000 cu. ft./hr. An overhead crane of 10 tons capacity with a 24 ft. lift serves the entire area.

Small laboratories, ranged along one side of the main laboratory, are provided for the preparation of metallurgical specimens, for microscopy and photographic work, for hardness testing, and for gas analysis. Instruments available include an infra-red gas analyser, a Hersch oxygen analyser, Foxboro dewpoint measuring and control units, and a "moisture monitor" dewpoint instrument. A Vickers projection microscope and a wide range of temperature measurement and control instruments are also available.

The department, which employs some thirty people, of whom about half have technical qualifications in physics, metallurgy, chemical engineering, mechanical engineering or electrical engineering—is charged with the application of scientific principles to the investigation and development of all types of equipment manufactured by the company. The work includes the examination of basic design principles, the testing of specific components, the performance evaluation of prototype plant, and the testing and final adjustment of specialised production equipment to provide data for future design and for the instruction of field engineers.

The organisation has four major sections dealing,



Adjusting atmosphere seals at the discharge end of the new stainless steel strip annealing furnace.

respectively, with metallurgical processes, furnace design, gas plants and dryers, and the testing of prototype equipment. A certain amount of work is also undertaken for an associate company, Birlec-Efco (Melting), Ltd., and the staff is responsible for operating a pilot smelting plant for this company at Aldridge. There is also located at Aldridge a contract heat treatment department which has at is disposal a wide range of production heat treatment equipment which can be made available for experimental work where necessary.

#### Work in Progress

A broad outline of the type of work carried out by the research and development department has been given in the foregoing: the following brief references to some of the matters under investigation at the present time will fill in some of the details, and will illustrate the variety of the problems involved.

#### Bright Annealing Stainless Steel Strip

The bright annealing of stainless steel strip is carried

out at high temperature in an atmosphere of cracked ammonia, and conventional annealing equipment incorporates a heat resisting nickel-chromium alloy muffle to prevent contamination of the protective atmosphere. There are a number of drawbacks to this method. In the first place the muffle is expensive and for many applications will need to be replaced from time to time. In addition, the heating elements are necessarily fitted outside the muffle and their duty is thus made more arduous than would otherwise be the

In the laboratory an experimental furnace is in operation in which the metal muffle has been eliminated, so that the strip and the heating elements are in the same chamber. This development is based on the use of special refractories for the furnace chamber lining. In addition to their being non-porous, it is essential that

they should not react with the atmosphere at the high annealing temperature to give rise to such contaminants as water vapour. By this means the disadvantages characteristic of the conventional equipment are overcome.

The laboratory set-up incorporates strip handling gear and equipment for removing oil and other surface contamination before entry to the furnace. Experiments can be undertaken with strip up to 6 in. in width and work has been carried out with austenitic, ferritic and martensitic stainless steels, with completely satisfactory results in all cases. Extension of the new principle to other bright annealing applications is receiving consideration.

#### Radiant Tube Heating Units

Whereas the time-honoured method of preventing contact between the products of combustion and the furnace atmosphere is by means of a muffle, recent years have seen a growing use of radiant tube heaters in which the flame or other heat producing device is enclosed in a heat-resisting tube, the hot part of which is inside the furnace chamber and heats the work

located inside the furnace chamber and heats the work by radiation.

A 15 ft. oil-fired radiant tube is operating in a specially constructed test rig in the laboratory, objects of the work in progress including the investigation of factors affecting temperature distribution along the length of the tube, the identification of operating conditions likely to lead to soot deposition in the tube, the design of burner suited to radiant tube operation, and the collection of operating data.

A gas-fired radiant tube in which a proportion of the products of combustion is recirculated within the tube is undergoing development. The objects of this work are the reduction of temperature gradients along the length of the tube and the development of a gas-fired unit which is interchangeable with the standard Birlec electric-radiant tube heating element.

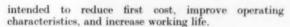
Development continues in the field of electric radiant tubes and two experimental units operating in the laboratory incorporate a number of novel features



Experimental rig for oil-fired radiant tube.



Setting-up a proportional controller on a large production exothermic generator.



#### Conveying Methods

A full-scale prototype shaker hearth furnace operating in the laboratory is a heavy duty unit which is intended to extend the scope of continuous hardening equipment up to the range of the larger cast link conveyor type of furnace. The principal feature of the new unit is a very much modified hearth design, aimed at reducing hearth distortion.

Also installed in the laboratory is a full-scale sealed quench carburising unit which includes the usual quenching facilities, but incorporates a number of simplifying features relating to a heating and quenching system and the associated work of handling through the furnace. In addition, it has been utilised as a means of subjecting unprotected heating elements of various designs to a process of life-testing under conditions closely approximating to those obtaining in gas carburising practice.

One of the problems associated with continuous furnaces operating in the production line is the amount of space they take up, and development work is in progress on a vertical pusher-type continuous annealing furnace in which the work passes up one side and down the other. The saving in floor space achieved by such a design is obviously considerable.

A current investigation of considerable interest concerns a novel method of conveying sheet material through a heat treatment furnace.

#### Furnace Atmospheres

The importance of atmosphere control in heat treatment plant is reflected in the amount of work in progress on the generation and purification of protective atmospheres. A prototype exothermic gas plant with flame-failure protection represents an attempt to reduce both cost and size of exothermic gas generators and to improve



Development of a vertical pusher annealing furnace.

operation. A gas/air mixture is combusted in a tunnel-type burner and the product gas is cooled by being passed through a curtain of water. The objects of the investigation include the collection of data regarding the design and operation of the burner, the demonstration of the efficiency of the direct water cooling unit, and the assessment of the quality of the product gas with regard to metallurgical applications. A number of commercially available flame-failure protection devices have been tested on the laboratory plant with a view to selecting the unit best suited for incorporation in the production generator.

Work is in progress on the development of improved catalysts for use in endothermic type gas generators, the experiments being carried out in a full-scale Birlec generator of conventional design.

Flame curtains are sometimes used to reduce the loss of atmosphere gas at furnace openings, and the design of an improved flame curtain burner and features promoting gas economy are being investigated.

Other work related to controlled atmosphere furnaces includes the development of improved high temperature circulating fans and an investigation into the rate at which oxygen will diffuse into a controlled atmosphere furnace against an outward flow of gas.

#### Removal of H2O and CO2 from Gases

A new series of adsorbent type dryers for compressed air and gases has recently been marketed. The essential design data were established and the final plant development and type testing were carried out in the laboratory, and examples of the smaller sizes in this range have been retained for experimental purposes.

Work is in progress on the further development of the

Birlec dehumidifying unit. Equipment has been constructed in the laboratory which simulates a section of a standard dryer, and experiments are planned in which the behaviour of different desiccants in this type of dryer will be studied. An appropriate technique and apparatus has also been developed for assessing the properties of desiccants on a comparative basis.

The company already markets a standard range of stripping units in which carbon dioxide is absorbed in an aqueous solution of mono-ethanolamine, but two possible alternative processes are under development in the laboratory. The first of these involves the use of a suitable adsorbent material, and is similar in operating principle to the conventional gas drying equipment containing activated alumina. This represents a considerable simplification in stripper design and operation when compared with the more usual mono-ethanolamine type unit. The equipment in the laboratory consists of a large rig in which the capacity of the adsorbent has been determined in relation to conditions of operation and reactivation.

The second process under development is that of the absorption of carbon dioxide in water at high pressure. An experimental unit has been erected in the laboratory

and it is intended that tests should commence in the near future.

Miscellaneous

A small high vacuum, high temperature unit is available in the laboratory to provide both process and design data on the application of vacuum techniques to such operations as annealing, stress relieving, brazing and sintering. Experiments are being made elsewhere in the laboratory to develop seals suitable for use on the ends of a high vacuum continuous strip annealing furnace.

In the carburising field, a bench-scale investigation is being initiated in which the high temperature carburising characteristics of various steels will be studied. This work is also intended to yield process data relating to high temperature carburising.

The associated company Birlec-Efco (Melting), Ltd., deals with induction units for heat treatment and melting, and with arc furnaces for melting and smelting. In addition to its operation of the pilot smelting plant at Aldridge, the research and development department also undertakes investigations on behalf of Birlefco. Among these is a study of the characteristics of power arcs and the possible methods of influencing their behaviour.

#### Aluminium for Architects

ON July 18th, at a large gathering of leading architects and representatives of the various sections of the building industry in the London region, The British Aluminium Co., Ltd., introduced its new British Aluminium Building Service, which represents a major step forward in the bringing together of the suppliers and manufacturers of aluminium products and the end-users of those products. Sir William Strath K.C.B., managing director of British Aluminium, who welcomed the architects on their arrival at the meeting, remarked, in introducing this new service, that it was appropriate that so soon after the Congress of the International Union of Architects, the headquarters building used by that Congress on the South Bank of the Thames should be the venue for launching a major scheme to enable British architects to make the fullest possible use of one of the "new materials and techniques," namely aluminium.

The manager primarily responsible for the planning of the Service, Mr. Neill Newsum, explained its operation and introduced Mr. Eric Bird, M.B.E., M.C., A.R.I.B.A., technical and research officer at the Building Centre. who commented on this new project from the user's point of view. In paying tribute to Mr. Bruce Martin, A.R.I.B.A., whose advice had been widely sought and taken, especially in compiling the special documentation, Mr. Newsum stressed that the British Aluminium Building Service had been planned and executed to a large extent by architects for architects. It is, of course, the architect who will lead the way in using and applying new materials and techniques in buildings. Thus the sponsors of this scheme have analysed the needs of the architectural profession and tried so to organise their service that it will provide the maximum possible aid to each individual architect and, in particular, to provide in a simple and direct manner the information he needs to obtain the right product or material for his designs.

In effect, the result has been a co-ordination and

correlation of a large mass of separate pieces of information in a single comprehensive manual, appropriately entitled "Aluminium for Architects." One copy of this book is being supplied free of charge to every architectural practice in the United Kingdom, and will be maintained constantly up-to-date as new developments occur in the future. This manual, although the most "visible" aspect of the British Aluminium Building Service, is, however, only the first part of it, for a comprehensive follow-up information service, organised on a regional basis, and in the closest collaboration with all the fabricators of aluminium building products, is provided. The information service is staffed by specialists and is available at all times to give further advice and help to architects and users of those products.

The presentation on July 18th was, in fact, the first of a series which will be held in the various regional centres over the next few weeks. Distribution of the manuals to all the architectural practices will follow the regional pattern immediately after each regional presentation; all practices are planned to have received their manuals at the latest by 31st October, 1961.

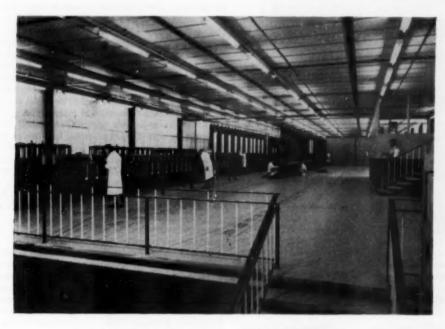
CLEARCALL communication equipment valued at £120,000 is to be supplied by the electronic apparatus division of Associated Electrical Industries, Ltd., for use throughout Richard, Thomas and Baldwin's new Spencer Works at Llanwern, Newport, Mon.

The offices and works of C. F. Casella and Co., Ltd., have been moved from Regent House, Fitzroy Square, London, W.1., to Regent House, Britannia Walk, London, N.1. (Tel.: CLErkenwell 8581 (6 lines)).

A New division of Associated Electrical Industries, Ltd., has been formed to manage the company's home district offices. Mr. V. S. Risoe, M.B.E., has been appointed general manager of the new division.

# The Creep Testing of Power Plant Steels

Electrical Research Association's New Laboratory



General view of the interior of the creep laboratory.

THE new laboratory for the creep testing of steel at elevated temperatures, built during the past year at the headquarters of the British Electrical and Allied Industries Research Association at Leatherhead, was opened on 6th July, by Dr. Charles Sykes, F.R.S., managing director of Thos. Firth & John Brown, Ltd. It is concerned with the improvement in the efficiency of generation of electricity, primarily in steam-driven turbo-alternator sets, and is the latest stage of a long investigation that has developed continuously over nearly forty years. In that period steam raising conditions at power stations have risen from 600 lb./sq. in. and 680° F. (360° C.) to 2,000 lb./sq. in. and 1,050° F. (565° C.), and the overall efficiency has consequently increased from under 20% to 34% in some cases.

This improvement has only been possible as the result of research on the high temperature properties of the steels used in the generating plant. The E.R.A. has been very actively associated with that research since 1930, when it set up a committee, upon which steel makers and turbine makers and users were represented, which sponsored the accurate investigation at the National Physical Laboratory of the high temperature behaviour of certain typical steels. As a result, a creeptesting laboratory was built at the N.P.L. and equipped with some seventy-five high accuracy creep testing machines. A number of steels was examined there and in addition members of the committee submitted appropriate experimental results obtained in their own

creep laboratories. It soon became evident that short-term testing was not sufficient, and periods of test had to be extended to times of the order of 20,000 hours or more. As knowledge of the properties of steels increased and steam conditions became more onerous, the number of special creep resisting alloys to be investigated also rose. The test programme thus expanded to such an extent that the laboratory at the N.P.L. could no longer cope with it, even though it had been reinforced by a further fifty machines installed in 1957 at the E.R.A. laboratories at Leatherhead.

The organisations represented on the committee decided to provide for an extended programme by means of a new laboratory at Leatherhead which would be devoted principally to long-time tests. As this plan coincided with a decision to build a new ERA office block at Cleeve Road, Leatherhead, it was suggested that the two should be built as one unit. This suggestion was adopted and the building opened by Dr. Sykes takes the form of an L-shaped structure, with the shorter limb in the form of a two-storey office block and the longer one a single-storey creep laboratory.

#### Laboratory Building

The requirements for this building presented a very interesting problem, in that it was necessary to limit the expenditure on building work whilst at the same time providing a high degree of insulation from climatic conditions outside and introducing some form of air

conditioning which would maintain a reasonable internal temperature and deal with the very high heat emission

from the creep furnaces.

It was decided to provide a clear floor span with lattice girders and a light structural steel frame, the roof to be of aluminium decking with cork insulation and three layers of felt covered with light coloured stone chippings to give some heat reflection. Additional insulation is provided by an Asbestolux ceiling lining with a layer of Rocksil immediately below the roof decking. Ribbed aluminium sheeting is used for the external walls, lined internally with Asbestolux sheeting, with additional insulation provided by a blanket of Rocksil hung in the cavity between the two. The bottom 4 ft. 6 in. of each side wall is in the form of an 11 in. cavity wall.

Since a full-scale refrigeration system would be too expensive to deal with the heat emission from the large number of furnaces to be installed, it was decided to use multiple air changes to dissipate the heat. By suspending a false ceiling under the bottom boom of the lattice girders, it was possible to use the whole of the area between the underside and the lattice as a duct, and so to ensure a slower air movement and provide an improved internal appearance. Five strips of continuous grilles are used in the ceiling to distribute the air, and concrete extraction ducts are provided in the floor along the line of creep machines with chequer plate covers bedded in mastic. An adjustible outlet is provided under each machine, together with a removable cover to allow for specimens from the furnaces to be removed.

As the building is windowless, to avoid direct solar



Ten-specimen rupture test machines.

gain, a high standard of artificial illumination is necessary, and this is provided by continuous strips of fluorescent lighting mounted immediately under the ceiling, supported by a continuous service trunking.

Eleven cubicles for testing to destruction simulated steam pipes under pressure abut the end wall of the laboratory. The total area including these is 12,725 sq. ft.

#### Testing Equipment

Since turbine makers base their rotor designs on  $0\cdot1\%$  creep strain in 100,000 hours, very small extensions have to be measured extremely accurately in creep testing, and this is best done with an optical extensometer attached to the test piece. The design of pipework and boilers, on the other hand, is based on the rupture properties of the components. Small changes in dimensions of the pipework are not necessarily serious, and hence only the time taken to rupture a specimen is needed, together with the amount of deformation at rupture. Another important factor in steam pipes is the strength of a butt-welded joint, and this involves both creep and rupture testing.

Additionally, the engineer needs information on the variation in properties from cast to cast of steels within a given specification. Thus a very large number of rupture test points is needed and can be most easily

provided for in multi-specimen machines.

The laboratory is equipped with fifty high sensitivity creep machines fitted with optical extensometers, fifty ten-specimen rupture machines and fifty dual purpose machines for either dial gauge creep measurements or three-specimen multi-rupture. One 15-ton creep machine is included for testing specimens from the full wall thickness of steam pipes. In addition to the creep and rupture machines, eleven rigs have been installed, designed to test tubes stressed by internal steam pressure while at high temperature. Four such rigs have been in operation for several years at Leatherhead and will be retained.

The creep testing machines are of the single lever type, and the high sensitivity units—the short machines on the left of the general view of the creep laboratory—are equipped with optical type extensometers developed by the N.P.L. The fifty rupture machines take the tenspecimens joined in series by split couplings in a 50 in. long furnace, the large extension during creep being taken up automatically by a motorised worm and wheel. The remaining fifty machines have 20 in. long furnaces and may be used for rupture testing three specimens in series, or for creep testing larger sized specimens using a low sensitivity dial gauge extensometer.

The furnace temperatures—of the order of 650° C.—must not deviate by more than 3° C. from their correct values. Such a change would be produced by a 0·4% change in applied voltage and the control circuit for each furnace comprises a resistance thermometer, an electronic control unit, and a saturable reactor designed to compensate for (a) fluctuations in mains voltage, and (b) changes

in ambient temperature.

The ideal position for the resistance thermometer—inside the furnace tube—is impracticable owing to the danger of breakage on specimen rupture. It is therefore inserted through the side of the furnace and rests tangentially against the furnace winding. Some doubt was felt as to whether this would be satisfactory if the winding consisted of widely spaced turns of wire, and the

furnaces are therefore wound with ribbon in closely spaced turns. A protective device is fitted to take care of failures of the controller which would cause the furnace

temperature to rise out of control.

Platinum/platinum-rhodium thermocouples attached to the specimens are used to measure the test tempera-To economise on the number of wires to be returned to the measuring desk, the large amount of platinum, and the large number of cold junctions that would otherwise be required, a system of switching close to the thermocouples was developed, in which all temperature measurements are made at a central desk. A selector on the desk actuates a switch close to the furnace which switches all the thermocouples from that furnace onto busbars of compensating wire. A second switch on the desk then selects the busbar corresponding to the thermocouple to be measured by the potentiometer. In this way only one cold junction is required for a set of busbars, and copper wire can be used between the cold junction and the measuring desk. To facilitate the setting up of the furnaces, the thermocouples can be plugged into a recording system and measured at the furnace with a portable potentiometer.

Any furnace can be connected to 90 V., 100 V. or 110 V. busbars, this range catering for tests at any temperature between 500° and 750° C. A potential point on the transformer winding that corresponds approximately to the middle of the furnace winding is earthed through a current limiting resistance, an ammeter and an alarm relay. This is believed to be sufficient protection against earth faults which, although if allowed to persist may cause a temperature rise which may spoil a test, are expected to arise only when connections are disturbed or by the changing of a specimen, and staff will be able to remedy it if their attention is drawn to its

existence by the alarm system.

The chance of a failure of the insulation between the furnace heating winding and the steel tube on which it is wound has been made negligible by isolating the tube from earth, and by adopting a low voltage. A test terminal connected to the steel tube enables the insulation between the winding and the tube to be tested with the voltmeter while in service.

As mentioned above, there are fifteen chambers at Leatherhead for carrying out bursting tests on steam tubing under pressure, eleven of them located at the end of the new laboratory. In the illustration of the interior of one of them, an electrically heated furnace is to be seen on the right. Accurate temperature control of the specimen within the furnace is achieved by having a five-zone winding and an electronic controller coupled to a platinum resistance thermometer. At the top of the chamber, on the wall, can be seen a remote-controlled electrically-actuated switch which enables the temperature of each of seven thermocouples to be read from the measuring desk. Pressures of 10,000 lb./sq. in. are used inside the pressure vessel, and at the end of most of the rupture tests the furnace itself is almost totally destroyed. The doors of the chamber are arranged to swing outwards when an explosion occurs, in order to relieve the pressure on the chamber: the roof is of light construction and is expandable.

#### **Financial Support**

The cost of building the new laboratory has been borne by the following industrial organisations: the



Interior of bursting test chamber.

Central Electricity Generating Board, the Water-Tube Boilermakers Association, the British Iron and Steel Research Association, Associated Electrical Industries, Ltd., English Electric Co., Ltd., General Electric Co., Ltd., C. A. Parsons & Co., Ltd., Aiton & Co., Ltd., Stewarts and Lloyds, Ltd., and Tube Investments, Ltd., with the addition of a capital grant from the Department of Scientific and Industrial Research.

#### Aluminium Aerials for Lighthouses

A LIGHTSHIP and a lighthouse off the Irish coast have been equipped with television sets: they are the South Rock Lightship off Ballywalter, County Down, and the Mew Island Lighthouse off Donaghadee, County Down. In order that the sets may function normally, special aluminium aerials have been constructed from light, tough alloys provided by The British Aluminium Co., Ltd. Their design and construction have presented difficult problems since the very exposed sitings have necessitated the fabrication of unusually strong mast structures. The lightship aerial will present an additional problem, since it must be able to swing independently of the ship's movements in order to maintain a constant bearing on the transmitting station. The masts were constructed by Telerection (N.I.), Ltd., of Belfast.

The board of the Glacier Metal Co., Ltd., announces that arrangements are well advanced for the formation of subsidiaries in South Africa and New Zealand which will manufacture plain bearings for those markets. Glacier will hold the major proportion of the equity capital—initially to be about £50,000 total in each case—and South African and New Zealand interests the remainder. Production is expected to start in 9-12 months' time,



General view showing work in progress on the foundations for the new E.S.C. works at Tinsley Park.

# English Steel Corporation Developments

Construction Work at Tinsley Park

New 80 ton Electric Arc Furnace

THE VISCOUNT KNOLLYS, G.C.M.G., M.B.E., D.F.C., chairman of English Steel Corporation and of its parent company, Vickers, Ltd., must surely be the most proficient member of the peerage in the art of pouring concrete. Quite recently he was to be seen on TV news topping-off the new skyscraper which is to house the Vickers group in London, and only a short time before, on 7th July, he inaugurated the construction work for English Steel Corporation's new Tinsley Park Works by pouring the first skip of concrete for part of the foundations of this new £26 million steel works, scheduled to be in operation by the last quarter of 1962.

#### **Tinsley Park Works**

This scheme, the first stage of a long-term E.S.C. plan and the largest works development ever undertaken within the Sheffield city boundaries, will include a melting shop with two 100 ton electric furnaces, a 42 in. blooming mill, a billet mill with two 32 in. stands, and bar mills. The new plant will increase English Steel's total capacity for the production of alloy and special carbon steels to about one million tons a year and billet and bar production will be trebled.

Already about 3 million tons of earth have been moved and consolidated into terraces on which the new buildings are to be erected. The foundations for the works are now being laid and steel erection will begin this year for the buildings, which will cover a floor area of more than 1 million sq. ft. The melting shop, designed to accommodate two 100 ton arc furnaces, with space provided initially for a third, will consist of three bays (charging, casting and mould preparation) in one building together with a separate scrap bay. The charging and casting bays are each designed to take 150 ton overhead cranes.

The blooming and billet mill building, which will have an overall length of 1,750 ft., will also include the soaking pits, treatment, warehousing and despatching facilities for blooms and billets. The 1,100 ft. long building for the bar mills will be of similar construction to that of the blooming and billet mills, thus preserving an architectural unity of design for the two groups of mill buildings.

#### New 80 ton Arc Furnace

Later in the day, at E.S.C.'s River Don Works, Lord Knollys commissioned the largest electric arc melting furnace in the United Kingdom for the production of special alloy steels. This furnace, which has a shell diameter of 21 ft. and an electrical rating of 20 MVA., is capable of melting an 80 ton charge in less than three hours.

The furnace had been designed and built by Birlec-Efco (Melting), Ltd., after careful consideration of English Steel Corporation's requirements. Power is supplied to the furnace by a 20 MVA. transformer of A.E.I. manufacture which has a resistance transfer onload tap-change mechanism especially suitable for arc furnace duty. A 10% auxiliary reactor arranged for on-load switching is also provided to limit power surges during the initial melting down period. The main switch employed is an oil circuit breaker, having a 750 MVA. rupturing capacity, manufactured by A. Reyrolle & Co., Ltd.

Specially designed water-cooled low-voltage flexible cables are used to carry current from the transformer to the furnace. During the melting period this current is approximately 28,200 A. per phase. The furnace transformer secondary delta is made on the electrode arms at the point of connection to the flexible cables. This arrangement is specially designed to give a minimum reactance and high furnace power factor, particularly when the electrode arms are displaced away from each other. Each electrode arm is counter-balanced by means of four pneumatic cylinders, enabling relatively small motors to achieve rapid accelerations and high speeds with a minimum of inertia. These motors are of special design to operate with a well-proved A.E.I. amplidyne electrode controller. The electrode diameter is 20 in.

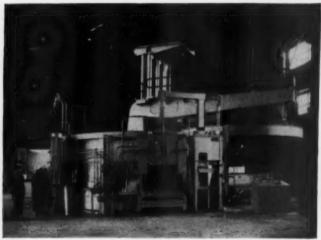
The furnace shell incorporates a new feature in that it is split horizontally in two halves just above the slag line. The upper half is detachable to enable the side wall brickwork to be replaced in the shortest possible time. To accomplish this, a spare upper half, which has



The casting bay in the electric furnace department at River Don Works, showing ladle in position over vacuum casting tank.

been previously bricked, can be lowered into position in a similar manner to the way an arc furnace roof is replaced in conventional practice,

This furnace has been designed for the production of special alloy steels for casting in vacuum. In conjunction with other melting furnaces it will enable English Steel Corporation to produce vacuum cast ingots up to 180 tons in weight. Ingots so produced will be extremely low in hydrogen content, and forgings made from these ingots will be particularly suitable for highly stressed



The new 80 ton Biriefco arc furnace at River Don Works with the roof swung away for charging purposes.

forgings for the electrical, chemical and general engineering industries, and for other high duty applications.

The new overhead travelling crane serving the electric melting and vacuum casting department is by Clyde Crane and Booth, Ltd., of Rodley, Leeds. It is equipped for handling the large ladles of molten metal from the electric furnace, and has a capacity of 200 tons on the main hoist. The auxiliary hoist has an independent crab running between the main girders and is of 50 tons capacity. The span of the crane is approximately 60 ft. and it runs on a gantry 59 ft. above the shop floor at rail level. Other features include Ward Leonard control, which, by converting the A.C. electric supply, enables the superior hoisting characteristics of D.C. motors to be utilised, and an enclosed driver's cabin with seated control unit and air filtration system.

#### United Steel buy Barrow Steel Works

The United Steel Cos., Ltd., have purchased Barrow Steel Works, Ltd., from the Iron and Steel Holding and Realisation Agency for the sum of £2,200,000. United Steel has managed this works since 1943 for a token remuneration. As a conventional steel-producing unit, the works has not been economic throughout this period. The re-rolling mills are not in themselves uneconomic, but their steel requirements are insufficient to support their own steelmaking furnaces, while the location of Barrow prevents the regular acquisition of billets from other sources of supply.

Continuous casting was considered to be the most promising form of small-scale billet production, based on local scrap supplies, and in the hope of converting the works into a profitable undertaking, a pilot continuous casting unit was installed in 1952. Since 1954, United Steel has borne the operating cost of the subsequent experimental work in view of its own interest in continuous casting developments.

The process is now working successfully, and two full-scale twin-strand continuous casting machines together with a 20-ton electric are furnace are being installed. This plant is expected to come into production in the

near future, and should provide billets at a cost which will enable the Barrow re-rolling mills to operate at a profit and so justify their continued existence. The scheme has been given the code name BARCON (Barrow Steel Works ARc Furnace CONtinuous Casting).

It is interesting to note that Barrow, which in 1877 had the largest blast furnace plant in the world and a total of 18 Bessemer converters, is now re-emerging as a pioneer. When BARCON is completed, it will be the first works in the world to be wholly dependent upon continuously cast steel for its rolling mills.

ALUMINIUM, LTD., have announced the formation of a company in the Federation of Malaya to be known as Alcan Malayan Aluminium Co., Ltd. The new company will build an aluminium rolling mill, the first in Malaya, to produce aluminium sheet, circles and building sheet. Initially the Malayan subsidiary will be wholly-owned by Aluminium, Ltd., who will finance the enterprise and furnish technology. Malay personnel will operate the plant and steps will be taken to train Malayan managerial staff. Located at Petaling Jaya, near Kuala Lumpur, construction is expected to start immediately. Estimated cost of the enterprise is over \$1,300,000.

# Lord Mills Opens New B.W.R.A. Laboratory

PORMALLY opening the British Welding Research Association's new 21,000 sq. ft. engineering laboratory at Abington on Wednesday 12th July, Lord Mills, a former Minister of Fuel and Power, told nine hundred guests that the research station, now capable of tackling any problem that may arise from the application of welding in industry, enjoys an international reputation and is unequalled anywhere in Europe—excepting Russia—or in the United States of America.

Quoting the example of one of this century's most revolutionary discoveries—that of radium—which was made in an old shed, Lord Mills said that if ever there was a chance of making discoveries as a result of working in tumble-down buildings, "the Association's staff has certainly had it." He was referring to the old stables and ex-army huts at Abington Hall, which, until the new building was commissioned, had housed the pressure vessel, resistance welding and brittle fracture researches. Although nothing as far reaching as the discovery of radium had come out of the Abington buildings, the results of the work done had made an important contribution to the efficiency, reliability, and safety of welded plant used in many different industries, and had substantially contributed to the high standard of welded design and fabrication in this country

After opening the laboratory, Lord Mills was presented with an inscribed paper knife by Mr. Robert Jenkins, chairman of the Association's council. The knife, which had been made at the research station, had a seam-welded stainless steel blade and an argon are welded aluminium bronze handle.

#### **Exhibition of Work in Progress**

The ceremony formed the start of a two day exhibition of research in progress which was attended by over twelve hundred industrialists, engineers, and representatives of scientific and professional organisations. In addition to the developments in conventional welding techniques and investigations shown, a friction welder built by the Association was demonstrated for the first time, and aroused a great deal of interest for its efficiency and simplicity. Although mild steel specimens, only, were welded during the exhibition, examples were shown of successful welds, in a variety of forms, in light alloys, stainless steels and titanium.

Other exhibits of particular interest were an electro slag welder, to be used to weld 4 in. to 6 in. steel specimens for testing in the Association's new 4,000 ton brittle fracture tensile test rig, and a demonstration of the work now being carried out on the study of arc characteristics. A high speed arc plasma jet, used among other things to investigate the phenomenon of metal transfer, was demonstrated, the arc being drawn out to a distance of 16–18 in. beyond the anode.

A recent innovation has been the integration of all departments in contact with members of the Association into one unit known as members' services. A detailed display showed how these various departments—design advisory service, library, liaison and publications—provided complementary services and assisted in providing a rapid dissemination of the Association's results in industry.



Mr. R. Jenkins, chairman of the B.W.R.A. council, presents Lord Mills with an engraved paper knife following the formal opening of the new laboratory: seated is Sir Charles Lillicrap, B.W.R.A. president. The bell seen in the photograph was taken from the clock tower of the old stables which, until recently, were used as research buildings: it was cast in 1712.

At the annual general meeting of B.W.R.A., held concurrently with the exhibition on the first day, Sir Charles S. Lillicrap was re-elected as president of the Association, and Messrs. D. J. W. Boag, C. H. Davy, T. McIver, R. B. Shepheard and Dr. T. W. F. Brown were re-elected to the council. Viscount Ednam and Mr. W. Muirhead Watson were additionally elected to the council.

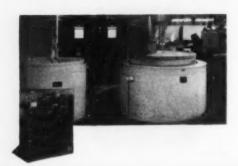
#### Welding Engineering 1962

The Institute of Welding is organising its first large scale exhibition at Buxton next year, in connection with its Spring Meeting there. Facilities in the Winter Gardens make it possible to expand the displays and demonstrations which have been features of recent Institute meetings into a specialised exhibition of welding and the allied processes and techniques-brazing, soldering, cutting, hard facing, metal spraying, manipulating, inspection, testing, and safety. The theme of the Spring Meeting is "Welding for Power Generation." Nearly thirty papers, including several from other countries, have been specially written for this meeting, the dates of which are 30th April to 4th May 1962. "Welding Engineering 1962," as the exhibition has been named, will illustrate the rapid progress of welding technology in this country, especially in meeting the challenge of modern power generation plant construction. There will be special sections devoted to research, education and publications.

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WE are indebted to the Jaguar Company for the above photographs of the new Model E and part of their Homocarb furnace installation. Homocarb with its twin advantages of forced circulation and exact temperature/time control has won world-wide recognition as will be seen from the following list of some important users.

Rolls-Royce, Austin, British Timken, B.S.A., Caterpillar Tractor, Humber, International Harvester, Singer, Sperry Gyroscope, Vauxhall, Morris, Jaguar, Villiers, Hoffman, Leyland, Rotol, Vandervell, etc., etc., are among hundreds of users of Homocarb.

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# **NEWS AND ANNOUNCEMENTS**

#### Forthcoming Lecture Courses

BATTERSEA COLLEGE OF TECHNOLOGY

Corrosion in the Electrical Power Industry—The Eighth Summer School on Corrosion, organised by the metallurgy department of the Battersea College of Technology in conjunction with the corrosion group of the Society of Chemical Industry, will be held from 25th to 28th September, 1961, and will deal with corrosion as it affects each stage in the generation, transmission and distribution of electric power. The fee for the course is £10 (inclusive of luncheon and morning and afternoon refreshment). Further particulars and enrolment forms may be obtained from The Secretary (Summer School), Battersea College of Technology, London, S.W.11.

BIRMINGHAM COLLEGE OF ADVANCED TECHNOLOGY

Two post graduate courses arranged by the department of metallurgy in the College of Advanced Technology, Birmingham, will be held during the autumn term.

Practical Implications of Metal Physics—A course of ten lectures given by leading authorities on consecutive Tuesday evenings at 6-30 p.m., commencing 10th October, 1961. The tutor for this course will be Dr. T. H. Richards.

Technology of Non-Ferrous Secondary Metals—A course of ten lectures given by members of the college staff and specialists from industry on consecutive Wednesday evenings, commencing 11th October, 1961. The tutor for this course will be Mr. K. A. Reynolds, and topics covered will include surveys of non-ferrous metals and alloys, sampling and sorting, melting and refining techniques, and commercial practice.

The fee for each of these courses is £2. Further particulars and forms of application may be obtained from the Bursar, College of Advanced Technology, Gosta Green, Birmingham, 4.

#### BOROUGH POLYTECHNIC

The division of metal science of the Borough Polytechnic has organised a series of special lecture courses for the winter term of the 1961-62 session.

Modern Developments in Non-destructive Testing of Metals—A course of six lectures given by specialists from industry on consecutive Wednesday evenings at 7 p.m., commencing 4th October, 1961. The fee for the course—intended for metallurgists, engineers and others interested in this field—is 10s.

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Recent Advances in Semi-Conductor Metallurgy—A course of six lectures given by Mr. A. S. Abrahams on consecutive Tuesday evenings at 7 p.m., commencing 24th October, 1961. The fee for the course—intended for metallurgists, physicists, electrical engineers and others engaged on research and development work in this field—is £1.

Corrosion and Protection of Buried Metal—A course of six lectures given by specialists from industry on consecutive Thursday evenings at 7 p.m., commencing 2nd November, 1961. The fee for the course—intended for metallurgists, chemists, civil engineers and others engaged in work in this field—is 10s.

Further particulars may be obtained from The Division of Metal Science, Borough Polytechnic, Borough Road, London S.E.I.

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#### ENFIELD TECHNICAL COLLEGE

Surface Treatment and Protection of Metals—A course of nine lectures given by authorities in the field, on consecutive Monday evenings at 7-30 p.m., commencing 15th January, 1961. Topics to be covered will include surface finish and properties, sub-surface structures and properties, electroplating and other methods of protection, and corrosion testing. The fee for the course is £1. Application forms and further details can be obtained from Mr. V. James, Metallurgy Section, Enfield Technical College, Queensway, Enfield, Middlesex.

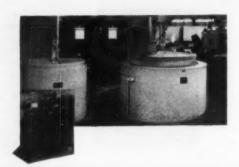
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THE Autumn Meeting of the Institute of Metals will be held in Brussels, by invitation of the Union des Industries de Métaux Non-Ferreux, from 18th to 22nd September. 1961. At the opening session in the Palais des Congrès. the Institute's Autumn Lecture on "The Non-Ferrous Metals Industry in a United Europe" will be delivered by Baron Snoy et d'Oppuers. Technical sessions will be held on the Tuesday and Thursday mornings when the papers will be related to the following general themes: deformation and fracture (Tuesday); and surface friction and lubrication in metal working, and corrosion (simultaneous sessions on Thursday). The remainder of the week will be taken up by visits to Belgian engineering and metallurgical works and a number of social functions which will include a reception at L'Hôtel de Ville, by invitation of the Bourgmestre de la Ville de Bruxelles, and a banquet and dance by invitation of the Institute's hosts at the Concert Noble.

#### Griffin & George Exhibitions

Two exhibitions at which modern laboratory equipment will be shown, are being staged by Griffin & George, Ltd., in Cardiff and Manchester. They will be held in the Welsh College of Advanced Technology, Cathays Park, Cardiff from 12th September to 14th September, inclusive, and at the Chorlton Town Hall, Cavendish Street,





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Manchester, from 18th September to 21st September, inclusive. Invitations are being sent by the company to all customers in the area, and enquiries should be addressed to the group's regional service centres at Birmingham (Mr. C. J. Passmore) and Manchester (Mr. D. F. Savage), respectively.

#### Second Pipes and Pipelines Exhibition

LAST year more than 30,000 miles of new gas and oil pipelines were constructed throughout the world. bringing the total up to nearly one million miles. In addition, many thousands of miles have been laid for the conveyance of other liquids, and even solids, and with the development of many new materials of construction, pipelines can now be used for highly corrosive liquids which a few years ago could not be handled in this way. It is not surprising, therefore, that considerable interest is being shown in the Second International Pipes. Pipelines, Pumps & Valves Exhibition and Convention to be held in April, 1962, at Earls Court. Over a hundred companies have already booked stand space, many of whom represent overseas companies, as well as prominent British manufacturers and construction organisations. A technical convention is to be held in the exhibition buildings at the same time, and full details of this as well as remaining available stand space can be obtained from The Organiser, Scientific Surveys Ltd., Exhibition Division, 97 Old Brompton Road, London S.W.7. (Telephone: KNIghtsbridge 9360).

#### Symposium on Corrosion in the Nuclear Energy Industry

The Société de Chemie Industrielle is organising on the 19th and 20th October, 1961 at the Maison de la Chemie in Paris, a symposium on "Corrosion in the Nuclear Energy Industry" with, as president of honour, M. F. Perrin, High Commissioner for Atomic Energy, and under the patronage of many eminent European personalities. The programme, which has been drawn up by a scientific committee under the chairmanship of M. Georges Chaudron, is particularly concerned with corrosion in fuel element sheath materials. Anyone desirous of participating should apply for an application form to Société de Chemie Industrielle, 28 rue Saint-Dominique, Paris, 7e, France.

#### Radiospectroscopy of Solids Conference

The Institute of Physics and The Physical Society and the British Radio Spectroscopy Group announce that they are jointly holding a two day conference on "Radiospectroscopy of Solids," in the department of physics of the University College of North Wales, Bangor, on 21st–22nd September, 1961. The Institute and Society's Guthrie lecture will be presented during the conference by Dr. D. Shoenberg, F.R.S., who will speak on: "The de Haas-van Alphen Effect and the Electronic Structure of Metals."

Residential accommodation will be available. Registration is necessary and the closing date for applications is 1st September, 1961. Details and application forms are available from the Administration Assistant, The Institute of Physics and The Physical Society, 47 Belgrave Square, London, S.W.1.

# International Conference on the Physics of Semi conductors

The Institute of Physics and the Physical Society, on behalf of the International Union of Pure and Applied Physics and the British National Committee for Physics, is arranging an international conference on "The Physics of Semiconductors," which will be held at the University of Exeter from 16th–20th July, 1962. The conference is planned to follow the previous sequence of conferences on the physics of semiconductors, which were held in Reading in 1950, Amsterdam in 1954, Garmisch in 1956, Rochester in 1958 and Prague in 1960.

Accommodation will be provided in halls of residence at the University. Further information regarding the conference may be obtained from the Administration Assistant, The Institute of Physics and The Physical Society, 47 Belgrave Square, London, S.W.1.

#### The Institute of Welding

MR. H. West, managing director of A.E.I. (Manchester), Ltd., was installed as president of the Institute of Welding for 1961–62 at the annual general meeting on 13th July. Two new vice-presidents also took office at this meeting: Mr. C. H. Davy, director of Babcock and Wilcox, Ltd., and Mr. L. Redshaw, deputy managing director of Vickers-Armstrongs (Shipbuilders), Ltd. The following have been elected to the council of the Institute: Mr. E. V. Beatson, Mr. M. Birkhead, Mr. V. W. Clack, Mr. A. Evitts, Mr. S. H. Griffiths, Mr. J. Hooper, Mr. J. McLean, and Dr. R. Weck.

#### Industrial Water and Effluents Group

A NEW subject group of the Society of Chemical Industry is being formed under the chairmanship of Dr. B. A. Southgate, director of the Water Pollution Research Laboratory of D.S.I.R. The group, to be known as the Industrial Water and Effluents Group, will be interested in all respects of the science and technology of treatment, conservation and economic use of water, and of the treatment and disposal of effluents. A programme of meetings is being arranged for the 1961/62 session, the first of which will take place on 1st November at the Royal Institution, 21 Albermarle Street, London, W.1. Anyone interested in becoming a member of the new group is invited to write to the General Secretary, Society of Chemical Industry, 14 Belgrave Square, S.W.1. or to the Industrial Water and Effluents Group Secretary, J. L. Hewson, Imperial Chemical House, Millbank, London, S.W.1. from whom further particulars and membership application forms may be obtained.

#### Safeguards in Foundries

REQUIREMENTS for the protection of workers engaged in the melting or founding of non-ferrous metals are proposed in the revised preliminary draft of the Non-Ferrous Metals (Melting and Founding) Regulations which have been issued by the Ministry of Labour. These regulations are based on the Report of the Joint Standing Committee on Safety, Health and Welfare Conditions in Non-Ferrous Foundries. The first preliminary draft of the regulations, which was entitled "The Non-Ferrous Foundries Regulations," published in November, 1959, contained a general invitation to make comments on the proposed regulations.

The new text has been prepared in the light of the numerous observations received, and of consultations held with organisations of employers, workers and interested parties. The major change has been the inclusion within the regulations of that part of the industry dealing with the melting and casting of metal for the production of non-ferrous ingots, billets, slabs, and other similar non-ferrous products, and a consequent amendment of the title to meet the revised scope of the regulations.

Copies of the draft regulations may be obtained from the Ministry of Labour, Safety, Health and Welfare Department (W.5), 19, St. James's Square, London, S.W.1. Any observations on them should be made in writing to the Ministry of Labour, 19, St. James's Square,

S.W.1. by 31st August.

#### News of Soviet Science and Technology

The Soviet Exhibition held at Earl's Court during July gave the British public the opportunity of inspecting the products of Soviet science and technology. A continuous watch on Russian technical progress is maintained by the Department of Scientific and Industrial Research, and reported in its Russian Translating Programme. Seventeen of the leading Russian technical journals are translated into English and published every month by British research associations and scientific societies for the Department. These provide up-to-the-minute information on Soviet progress in steel manufacture, castings, welding, rubber and plastics, production engineering, machines and tooling, instrument construction, textiles, wood processing, chemistry, biology and mathematics.

The Department also sponsors the translation and publication of Russian technical books, and each year places on sale translations of hundreds of technical articles. These translations are obtainable from the new National Lending Library for Science and Technology, which also has available on loan a further 20,000 individual translations. Further details may be obtained from The Director, National Lending Library for Science and Technology, Boston Spa, Yorkshire. (Tel: Boston

Spa 2031.)

#### **Export Orders for Wild-Barfield**

RECENTLY, a considerable number of export orders has been received by Wild-Barfield Electric Furnaces, Ltd., of Watford, Herts. They have come from at least ten different countries ranging from Austria to Australia

and Finland to France, and include Japan.

Among the interesting furnaces called for are vacuum induction and resistance heated equipments for Sweden and the International Atomic Energy Agency in Austria and a vertical pit-type gas carburising equipment for Finland. A large mesh belt conveyor furnace and an ACE sealed quench furnace will be going to swell the not inconsiderable amount of Wild-Barfield equipment in Holland, while an electron beam welding unit will be started up in Belgium as well as a pit-type vacuum furnace of the internal element type.

furnace of the internal element type.

Billet heating furnaces for India, Tensometer furnaces for Australia and high-speed steel hardening equipment for France are among others on the list, whilst a significant export is that of a mains frequency induction heated aluminium holding furnace to Japan, where it

will be employed in the manufacture of low-pressure die cast light alloy components for use in the automobile industry.

The continuing receipt of these and other export orders enables Wild-Barfield to maintain the proportion of export business at approximately 30% of the whole, a fair figure for heat treatment and kindred equipment, but one for which strenuous efforts are being made always to improve

#### Contract Heat Treatment

A NEW company, Automobile and General Heat-Treatments, Ltd., has recently been formed with Mr. D. T. Ellis and Mr. A. H. Preedy as directors. The company is engaged solely on heat treatment to customers requirements, the shaker hearth equipment installed being specially suited to the nut and bolt trade. Already working day and night shifts, the company is shortly leaving its premises at Woden Road West, Wednesbury, Staffs. for larger ones with a greater range of furnace equipment to carry out a wider variety of heat treatment operations.

#### Personal News

Mr. J. A. V. Watson, O.B.E., will be appointed assistant managing director of Castrol Industrial, Ltd., upon the retirement of Mr. W. F. List on 1st January, 1962. Mr. Watson's appointment as an assistant managing director of the parent company, Castrol Ltd., to take effect on the same date, has already been announced.

On Thursday, 6th July, Mr. H. F. Sherbourne, M.C., managing director of Yorkshire Imperial Metals, Ltd., was appointed the new president of the British Non-Ferrous Metals Federation in succession to Mr. W. W. Dolton, managing director of the Delta group of companies.

Mr. R. A. Nicholson, steel sales manager of Sanderson Brothers and Newbould, Ltd., Sheffield, has retired after an unbroken spell of fifty-one years' service. Before becoming steel sales manager, Mr. Nicholson was production manager of the steel department.

SIR Hamish MacLaren K.B.E., C.B., D.F.C., former director of electrical engineering at the Admiralty, has been appointed a consultant to Associated Electrical Industries, Ltd. He will advise on all matters concerning electrical equipment for ships.

To improve the internal efficiency of the Hilger & Watts group, the Infra Red Development Co., Ltd., have strengthened their board of directors by the election of Mr. W. Bamford and Mr. B. E. Newman: Mr. J. B. Johnston has been appointed secretary. As a further step towards closer integration, Mr. P. Walduck, the company's sales manager, has transferred his sales office to 98 St. Pancras Way, London, N.W.1., where it will become part of the process-control division of Hilger & Watts, Ltd.

AFTER fifty-one years' service with Crookes, Roberts & Co., Ltd., manufacturers of heater and refiner bars and a member of Tempered Group, Ltd., Mr. A. Parsons, director and general manager, retired at the end of July.

Mr. D. Parkinson has joined the public relations division of the Basil Butler Co., Ltd., the advertising

agency from the public relations department of Handley Page, Ltd.

Dr. J. E. Holmstrom has joined Pergamon Press as secretary general of the scientific conference centre established recently to assist the organisers of international meetings in their preparatory work and to arrange all necessary services, afterwards ensuring rapid publication of the proceedings. In addition he will edit certain special categories of publications.

VICKERS, LTD., have established a new category of head office executives—principal officers of Vickers, Ltd., and have made the following appointments: comptroller—MR. J. H. ROBBIE, controller of research—DR. C. F. BAREFORD (who retains his appointment as managing director of Vickers Research, Ltd.), controller of contracts—MR. N. H. JACKSON, and controller of forward development—MR. J. G. LLOYD. Each of these officers will be responsible to a director of Vickers, Ltd.

Vickers-Armstrongs (Engineers), Ltd., have announced the appointment of Lt. Cmdr. R. B. Lakin, D.S.O., D.S.C., as assistant managing director (northern works) and of Mr. S. P. Woodley, M.B.E., as assistant managing director (southern works), in addition to their existing duties. Mr. J. A. Killick has been appointed a director and general manager, Crayford, Dartford and Thames works, in succession to Mr. J. G. Lloyd, who has been appointed a principal officer of Vickers, Ltd.

VICKERS-ARMSTRONGS (SHIPBUILDERS), LTD., have announced the appointment of Mr. L. Redshaw as deputy managing director, located at the company's headquarters on Tyneside. Mr. R. M. Nicholson has been appointed a director and general manager, Barrow shippyard, and Mr. W. Parnell a special director and shipbuilding manager, Barrow, in succession to Mr. Nicholson.

IOCO, LTD., announce the appointment of Mr. H. F. Judd, as managing director in place of Mr. J. A. Killick, who has relinquished his seat on the board on his appointment as a director of Vickers-Armstrongs (Engineers), Ltd., and general manager Crayford, Dartford and Thames works.

MR. H. C. H. MATTHEWS has been appointed a technical director of Pantak, Ltd., of Vale Road, Windsor, Berks. Due to pressure of other business commitments, SIR Peter Roberts, Bart., M.P., has resigned from the board of Staveley Industries, Ltd.

P. A. May & Partners, Ltd., have appointed Mr. E. P. Collins as director in charge of the agency's industrial accounts. For the past fourteen years Mr. Collins has been manager of group advertising for Morgan Crucible.

Dr. J. T. Norton, professor in the department of metallurgy of the Massachusetts Institute of Technology, received the Plansee Plaque awarded at the Fourth International Plansee Seminar held in Reutte, Tyrol, Austria, from 20th to 24th June.

Mr. J. C. Shapley, works manager at Hoover, Ltd.'s factory at Perivale, Middlesex, who recently completed twenty-five years' service with his company, has been appointed to the board of Hoover (Electric Motors), Ltd., Cambuslang, Lanarkshire.

TEMPERED GROUP, LTD., of Sheffield announce two new appointments to the board of directors of Henry Rossell & Co., Ltd., one of the companies recently acquired by

the group. They are Mr. J. E. Gibb, until now establishment manager of Tempered Spring Co., Ltd., another member of the group, and Mr. K. G. Settle, secretary/accountant of Rossell's.

The United Steel Cos., Ltd., announce that Mr. S. Beer, who has been head of the company's department of operational research and cybernetics since its inception in 1957, has resigned to become managing director of a new international firm of operational research consultants based in London. He is succeeded by Mr. D. Owen, previously assistant head of the department.

The British Steel Castings Research Association, Sheffield, announces that Mr. W. S. Scott, managing director of The Darlington Forge, Ltd., has been elected chairman of the council in succession to Dr. R. Hunter, deputy managing director of The Clyde Alloy Steel Co., Ltd., and that Dr. C. J. Dadswell, managing director of the English Steel Castings Corporation, Ltd., has succeeded Mr. W. S. Scott as vice-chairman of the council of the Association.

Mr. R. K. Cuthbertson, W. S., has been appointed chairman of R. B. Tennent, Ltd., rollmakers of Coatbridge, in succession to the late Mr. James Tennent.

FOLLOWING the appointment of Mr. H. B. Morris to the board of directors, Wickman, Ltd., announce the appointment of Mr. H. Andrews as secretary and Mr. H. L. Hudson as chief accountant.

MR. D. F. CAMPBELL, chairman of Davy-Ashmore, Ltd., has announced his intention to retire from the board at the conclusion of the forthcoming annual general meeting in September. Mr. Campbell joined the board of Davy Brothers, Ltd., in 1935 and became chairman of its successor, Davy-United, in 1944. Upon the merger with Power-Gas Corporation in 1960, Mr. Campbell agreed to continue as chairman of Davy-Ashmore, Ltd., during the transition period. The Board of Davy-Ashmore, Ltd., has designated Mr. M. A. FIENNES to succeed Mr. Campbell as chairman and Mr. L. H. Downs as vice-chairman. Mr. Fiennes will continue as group managing director of Davy-Ashmore, Ltd. Whilst remaining chairman of Davy and United Engineering Co., Ltd., Mr. Fiennes has now relinquished his appointment as managing director of that company and MR. M. F. Dowding has been appointed in his place. Mr. Dowding is already a director and was formerly general manager of the machinery division. Major W. R. Brown, a former chairman of The Power-Gas Corporation, Ltd., retired from that board prior to the merger in 1960, but joined the board of Davy-Ashmore to assist during the transition period. He also will retire at the conclusion of the annual general meeting, but will remain a director of Ashmore, Benson, Pease & Co., Ltd.

The George Cohen 600 Group, Ltd., announce that Mr. J. P. Bolton has been appointed managing director of their Australian subsidiary, George Cohen Sons & Co. (Australia) Pty. Ltd., of Sydney. He succeeds the late Mr. C. A. Bell.

MR. P. C. FRYER has been appointed to the staff of the Lead Development Association, where he will be principally engaged in development work for the lead industry in the battery field. Mr. Fryer is a metallurgist and prior to joining the L.D.A. was on the staff of the Morgan Crucible Co., Ltd.

# RECENT DEVELOPMENTS

MATERIALS : PROCESSES : EQUIPMENT

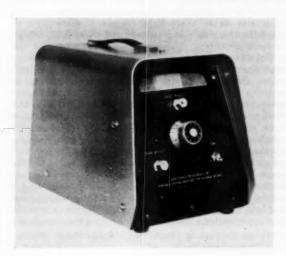
#### Simple Nuclear Health Monitor

Now that more and more organisations are involved in operations where radioactive materials are handled, a need has arisen for a small inexpensive monitor to check personnel's hands for contamination before they leave the premises. The E.M.I. single hand monitor has been specially designed to cater for the needs of the smaller laboratory or other establishment where there is a risk of radioactive contamination. It can check five people's hands every minute. Transistors are used to give a high degree of accuracy, and both audio and visual alarms are incorporated. The durable casing is made from Cellobond polyester resin reinforced with glass fibre. This is an ideal material for making prototypes and small batches as its use does not necessitate excessive tooling costs.

E.M.I. Electronics, Ltd., Hayes, Middlesex.

#### Portable Oxygen Analyser

SERVOMEX CONTROLS, LTD., who manufacture the Distillers Company's industrial oxygen analyser, have now developed a portable instrument utilising the same magnetic susceptibility measuring cell. The type DCL 101 portable analyser is a battery operated device which can be regarded as an alternative to the Orsat apparatus for the determination of oxygen anywhere in the range 0-100% to an accuracy of plus or minus 0.1%. Operation is simple and as the device is linear, calibration at two points is sufficient to standardise over the whole scale. The zero is highly stable and a check of the span using air as a test gas is sufficient for most normal uses. The analyser may be used with static samples or with a continuously flowing sample at rates up to 150 ml./min. The sample must be filtered and cooled sufficiently to remove condensate, but there are otherwise few restric-



tions. The measuring cell is particularly tolerant of variation of sample components other than oxygen, and it is not in any way affected by thermal conductivity or density variations.

Experience with the industrial analyser has shown that the DCL measuring cell is extremely robust. It contains a very light dumb-bell shaped test body suspended by a platinum ribbon in a non-uniform magnetic field. The torque on the test body produced by the presence of oxygen in the measuring cell is balanced by passing current through a coil wound on the dumb-bell. The balance condition is observed by means of a light spot reflected on to a scale and is obtained by adjusting a calibrated source potentiometer. The read-out is taken direct from a linearly calibrated ten-turn dial.

The DCL 101 portable oxygen analyser is used for routine oxygen determination and is a potential time saver, being quick, clean, reliable and simple to use. It is strongly built and can be made ready for use within seconds. It is 13 in. long  $\times$  8½ in. wide  $\times$  9½ in. high, and weighs only 18 lb.

Servomex Controls, Ltd., Chemical Instrument Division, Crowborough, Sussex.

#### High Conductivity Spring Alloy

Johnson Matthey & Co., Ltd., announce the development of Mallory 53, a nickel-silicon-copper alloy that fulfils the need for a spring material with mechanical properties at least equal to those of the conventional phosphor bronzes and brasses, but with appreciably higher electrical conductivity. The need for a spring material with such characteristics has been felt for some time by designers of electrical equipment, especially those involved in the production of miniaturised components.

Because of the exceptionally high electrical conductivity of the alloy, 40–45% I.A.C.S., it is possible for springs of small section to carry appreciably larger currents when made in Mallory 53 alloy than when made in phosphor bronze. Advantage can be taken of this characteristic to increase the current rating of existing designs of springs. All spring forming operations can be applied to Mallory 53 after heat treatment and hence the alloy is supplied in the precipitation hardened condition requiring no further heat treatment by the user.

A data sheet, 1300: 332, giving details of the characteristics of the alloy and the forms in which it is supplied, is available on request.

Johnson, Matthey & Co., Ltd., 73-83 Hatton Garden, London, E.C.1.

#### Tri-X Pan Professional Roll Film

KODAK, LTD., recently introduced the new Kodak size 120 Tri-X Pan professional roll film. This is an allpurpose film, intended for every assignment and for all industrial, commercial and professional black-and-white photography, as well as being specially suited to the needs of the press photographer. It offers the following advantages: (1) a new emulsion which gives shorter development and drying times, plus finer grain combined with moderate contrast, wide exposure and development latitude, and well-balanced colour sensitivity; (2) edgenumbered frames: each frame on each roll is numbered so that pictures can be identified immediately: and (3) each press-open pack contains ten foil-wrapped rolls of Tri-X Professional film: the pack slips neatly into pocket or equipment bag and can be opened in a second.

Kodak, Ltd., Kingsway, London, W.C.2.

#### Strippable Coating

SWALE CHEMICALS, LTD., announce the development of an easily strippable protective vinyl coating Type KP/100 for metal and glass. The material can be applied by brushing, spraying, dipping or flow coating. Used primarily to protect the surface of polished metal or plate glass, KP/100 is also suitable as a substitute for strippable tapes in painting and building construction. Tough enough to withstand abrasion, KP/100 is highly flexible and can be stripped off as a continuous film. It is resistant to moisture, common solvents, acids and alkali, oil, detergents, plaster and mortar. Due to its high film strength, KP/100 is said to be most economical in use.

Swale Chemicals, Ltd., 53 Park Hill Road, Croydon, Surrey.

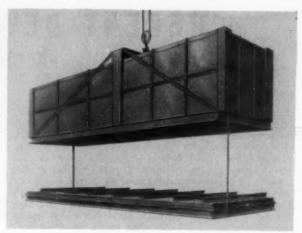
#### Lightweight Bell-Type Furnace

A NEW lightweight bell-type furnace, embodying a low thermal mass form of construction, has been specifically designed by G.W.B. Furnaces, Ltd., in conjunction with engineers of the South of Scotland Electricity Board, to achieve a predetermined heating and cooling cycle, while at the same time keeping its weight, price and running costs to a practicable minimum for the large physical proportions involved.

Although this furnace is also suitable for annealing and normalising, it is primarily designed for the stress relieving of heavy charges such as: heavy welded fabrications, cast iron bedplates, pressure vessels and boiler shells. Ideally, these types of charge should be stressed relieved in one piece to obtain the required uniformity, and because of the large physical size which normally applies, G.W.B. produce correspondingly large furnace equipment.

The heating bell casing has a sturdy fabricated mild steel structure, incorporating a main carrying frame of rolled steel sections with a single point lifting eye. This casing is lined with low thermal mass insulation embodying specially moulded semi-refractory element support bricks. Nickel-chromium (80–20) heating elements, in coil form, are set in the sidewalls and roof of the chamber.

Working with this bell heating unit is a charge supporting base, comprising a mild steel structure, adequately insulated, and topped with semi-refractory brickwork. Additional heating elements are accommodated in grooves provided in this surface refractory. Charge support members are provided, carried on mild steel posts which pass through the hearth insulation so that the charge load bears directly on the steel base structure.



To ensure correct alignment of the bell when hoisting or lowering, guide posts are fitted on opposing corners of the base.

The furnace rating is arranged in an appropriate number of zones dependent upon the chamber size, and the process cycle is governed by a programme controller. Permanent records of each treatment are provided by a supplementary recording instrument. All essential ancillary electric equipment is included, comprising switchgear with main air circuit breaker; "pick-up" contactor gear which ensures that the heating elements are automatically energised when the bell is lowered into position; and all necessary protective devices.

G.W.B. Furnaces, Ltd., Dibdale Works, Dudley, Worcestershire.

#### Spray Gun and Nozzles

ALFRED BULLOWS & SONS, LTD., announce the introduction of a new spray gun and a completely new range of FF fine finishing nozzles for use with the Graco Airless Hydra-Spray equipment, for which they are sole United Kingdom distributors. Designed to increase efficiency and reduce operator fatigue, the spray gun is known as the Hydra-Spray Golden gun. It is a lightweight spray gun whose new rotary-action packing eliminates leaks. Very light trigger pressure gives an immediate, precise spray pattern and there is no "lag" to cause spitting.

The advantages claimed for these new FF nozzles are that fine finishes can now be applied with feathered edges, thus enabling passes to be lapped; lower air pressure is required to operate the Hydra-Spray pressurising pump than was previously necessary; and, finally, the FF nozzles can apply thinner coatings than have so far been possible. There are seventeen FF nozzles, covering the complete range of fine finishing applications. The gun is equally efficient for high volume fine finishing or protective coating work, and the single hose enters the gun handle for improved balance; furthermore, coupling of the hose to the gun is by a special swivel attachment which gives the operator more freedom with less fatigue. A further feature is that the gun incorporates a tungsten carbide fluid valve and seat.

Alfred Bullows & Sons, Ltd., Long Street, Walsall, Staffordshire.

# CURRENT LITERATURE

#### **Book Notice**

BRITISH AND FOREIGN SPECIFICATIONS FOR STEEL CASTINGS—PART 2

Paper covers, 10 in.  $\times$  8 in., pp. xiii + 29. Published by the British Steel Castings Research Association, East Bank Road, Sheffield 2. 40s, postage paid. \$7.50 in United States and Canada.

SINCE its publication in December 1959, Part 1 of "British and Foreign Specifications for Steel Castings" has proved itself a "best-seller," orders having been received not only from the British steelfoundry industry and its customers, but from industrial concerns, organisations, institutions, librarians, and private individuals in all industrial countries. A measure of its usefulness is proved by the fact that in many instances. Part 2 has also been ordered, in advance of publication. Part 1 presented in summary form the main requirements for steel castings of the national standardising organisations in Austria, Belgium, France Germany (Western and Eastern), Great Britain, Sweden, Switzerland, and the United States. In addition lists were provided of standards current in Czechoslovakia, Holland and Italy. Part 2, like the first part, provides a "guide-book" to the identification of oversea standard specifications for steel castings and their comparison with British. In this case, the countries concerned are Australia. Canada, Denmark, India, Japan, New Zealand, South Africa and the U.S.S.R. It will be seen, therefore, that Parts 1 and 2 together embrace all countries of the Commonwealth, of the Common Market, and of the European Free Trade Association that issue national specifications for steel castings. Of countries outside these groupings account has been taken of the United States in Part 1 and of Japan and the U.S.S.R. in Part 2. The information given in Part 2 is that available on March 31st 1961.

#### Trade Publications

Rocol Molytherm Grease has been designed for continuous lubrication service at temperatures above 350° F; it withstands long periods above 450° F. and short periods up to 550° F. Based on a high flash point oil and a non-soap thickener which is unaffected by heat or chemicals, Molytherm Grease contains molybdenum disulphide. Publication No. 17, available from Rocol Ltd., Rocol House, Swillington, Leeds, describes this new grease and lists successful high temperature achievements in the metallurgical, chemical, engineering, glass and textile industries during its twelve-month development period.

A RECENT technical data sheet issued by Croda, Ltd., of Cowick Hall, Snaith, Goole, deals with G.P.2587, a quick-drying black dewatering rust-preventive which leaves a smooth, hard film on metal surfaces. It is composed of specially plasticised bituminous materials, dewatering agents, drying oils, and metallic soaps dissolved in quick drying solvents. It contains no abrasive substances and is entirely free from water and corrosive matter. It is designed to remove water from articles such as machined parts and to provide a rust preventive

film which will protect metal surfaces for long periods during direct outside exposure. It can be applied by dipping, spraying, brushing, swabbing or injection.

Johnson Matthey & Co., Ltd., have issued a new series of data sheets describing their range of small-bore tubes and tube products. Tubes to fine limits are available principally in non-ferrous metals and alloys, although grades of nickel-iron alloy are supplied, as well as tubes in precious metals for specialised applications. The data sheets describe Johnson Matthey tubes in terms of end uses, for example Bourdon tube, capillary tube, restrictor tube, instrument pointer tube, tube for applications in electronics, and miscellaneous base-metal tubes. Sets of data sheets and further information are available on application to the company at 73-83 Hatton Garden, London, E.C.I.

We have received from Old Park Engineering, Ltd., an illustrated brochure entitled "Welded Steel Fabrications" which describes the company's activities. Facilities are available for handling large units, the lifting capacity being 40 tons, and for vessels and other similar types of work, automatic equipment for welding with inert gases and the submerged are process have been installed. Other facilities are available for forming and machining fabrications both before and after welding. A stress relieving furnace is also available and the company, being a member of the Tube Investments Group, is backed by the resources of the Tube Investments research laboratories at Cambridge and the Tube Investments technological centre at Walsall.

THE Engineering Group of The General Electric Co., Ltd., has recently revised Technical Description No. 277 which deals with high torque, high slip and high reactance squirrel cage motors. High torque motors are substantially the same as standard squirrel cage motors with the exception that the rotor has two separate squirrel cage windings. By adjustment of the values of the resistances of the two windings, different speed-torque characteristics can be obtained. High slip motors are employed where the load varies very rapidly, sometimes to the extent of consisting of a series of sharp peaks with periods of light load, examples being shears, presses and hammers. G.E.C. has available a range of high slip motors for the types of drives mentioned above with outputs as high as 200 h.p. High reactance motors are available from G.E.C. for direct-on-line starting: this type of motor is especially suitable for drives in which inertia is high. A table included in T.D. No. 277 shows the diversity of drives for which G.E.C. high reactance motors have been supplied, and indicates the range of horsepowers, speeds and voltages of these motors,

In the many instances where straight mild steels are used for normal structural purposes, protection against atmospheric corrosion is often a major problem. At a very slight increase in cost the use of copper-bearing structural steels can provide considerable advantages in this respect. In a new C.D.A. publication (No. 61), entitled "Copper-bearing Steels for Structural Purposes," these advantages and the various properties of "copper-steels" are briefly summarised. It is shown that there are no associated difficulties in the use of copper steels

and that their ease of handling and manipulation is equal to that of the straight mild steels. Copies are available from the Association at 55 South Audley Street, London, W.I.

Johnson Matthey have issued the first data sheets of a revised and enlarged series describing products for the electrodeposition of the noble metals: silver, gold, palladium, rhodium, and platinum. Sheets issued deal with the types of high-purity silver anodes available for vat and barrel plating under a wide range of conditions. The acid hard gold method of plating printed circuits, bright gold plating, and the Galvorie immersion process for decorative uses are covered, and also palladium plating and the recently introduced DNS platinum process, which produces bright, heavy, and coherent deposits. Data sheets in preparation will give details of new rhodium plating methods and the well-established range of Johnson Matthey gold and silver salts.

The extraction of carbon dioxide from waste flue gas can now be accomplished by means of a new plant recently developed by the Incandescent Heat Co., Ltd. A reprint issued by the company's gas atmospheres division describes how this is done, and points out the advantage for foundries, breweries, refineries, and many other industries dependent on CO<sub>2</sub> as an indispensable part of the process.

"Corrosion Resistance of Hot Galvanising," the first in a new series of technical booklets to be issued by the Hot Dip Galvanisers' Association, is intended to give engineers, architects and designers up-to-date and accurate information on the performance of hot galvanised coatings in various environments—in contact with fresh and sea water, with oils, chemicals, building materials, in agricultural conditions and underground. The advantage of using heavy coatings to obtain more complete protection against rust is stressed. Copies may be obtained from the Hot Dip Galvanisers' Association, 34 Berkeley Square, London, W.1.

A NEW publication produced by Henry Wiggin & Co., Ltd., covers the selection of the most efficient materials for use in the handling equipment of the pickling processes used for the removal of surface oxide from hot worked or heat treated metals. The choice of material for the equipment and its design are the two aspects of the process on which this publication offers advice.

RAMAO—the subject of a recent publication by General Refractories, Ltd.—is a high magnesia ramming compound specially designed for the working bottoms of open hearth and electric arc steel melting furnaces. This material contains 90–92% magnesia with small amounts of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and CaO and has a maximum service temperature of 1,750° C. In addition to a table of typical physical properties, details are given of the way in which it should be used in installation and repair work.

We have received from Monometer Manufacturing Co., Ltd., Savoy House, 115-116 Strand, London, W.C.2., an illustrated leaflet describing the various types of melting furnace made by the company. These range from rotary furnaces for melting special irons at temperatures up to 1,650° C. to rotary furnaces for melting aluminium alloys at much lower temperatures, and from basin crucible tilting furnaces to dry hearth melting furnaces for use as bale-out units for aluminium die

casters. Other products featured include valve-outlet melting furnaces for white metals and type metal alloys, and hot metal receivers for the foundry industry.

"NICKEL IN S.S. Canberra" illustrates the thousandand-one applications of nickel-containing materials in
one specific case history. The Canberra is a revolutionary
vessel incorporating the latest thinking in architecture
and marine engineering. In breaking with many
traditions she is likely to set a new pattern in marine
design, and this publication describes some of the
interesting new features of the Canberra, and shows in
this the most modern vessel of its day how an established
and versatile metal—nickel—plays its vital part. This
publication is obtainable free on request from the
publicity department, The International Nickel Company
(Mond), Ltd.

WE have received from W. Canning & Co., Ltd., Birming-ham, the second number of *The Canning Journal* in which are featured extracts from two recent lectures presented by members of the staff on plating shop layout and installation, and plating shop effluent treatment, respectively. Additionally, there are sections dealing with recent developments in decorative chrome nickel plating, in automatic polishing, and in cycle rim plating. There is also a review of processes and equipment in the plating field and some illustrations of Canning installations.

Considerable interest has been created by articles in Durez Shell Molding News on the latest shell moulding procedures, new items of equipment, and practical experiences with the process in U.S. foundries. This publication is the organ of the Durez Plastics Division of Hooker Chemical Corporation and arrangements for copies to be sent directly from the United States, free of charge, can be made on application to the sole United Kingdom distributors, Omni (London), Ltd., 35 Dover Street, London, W.1.

The important part played by copper in the printing of all types of fabrics is illustrated in an article in the Summer 1961 issue of Copper, the journal of the Copper Development Association. Examples of the ubiquitous nature of copper are provided by other articles on its use in cross channel cables, in plumbing, in fireplaces and as a temporary protective on ferrous wire. Although the impact extrusion process has been used for many years for manufacturing thin-wall containers, many engineering designers are unaware of the advantages afforded by the process for the manufacture of certain components in copper and some of its alloys: an article aimed at remedying this situation describes the process and its application to copper alloys.

The "Buhova Trade Letter," published annually by the Bureau voor Handelsinlichtingen (Commercial Intelligence Office), Amsterdam C. (Holland), 16 Oudelrugsteeg, contains a large number of names and addresses of industrial and commercial firms in the Netherlands interested in trade relations with firms abroad. The entries are indexed according to product and any United Kingdom firms interested in business relations with Dutch firms should apply to the Bureau for a free copy.

We have received from Negretti & Zambra, Ltd., a copy of the revised and up-to-date version of publication S.109 which lists and describes Negretti & Zambra sales and service overseas.



Wild-Barfield A.H.F. equipment is used by Wolf Electric Tools, Ltd., (manufacturers of the well-known Wolfcub drills etc.), for the hardening and tempering of small gears, shafts and pinions. Many other industrial concerns have found that Wild-Barfield A.H.F. induction heating speeds production, saves space and offers savings all along the line. Our engineers will be glad to supply further details and explain how Wild-Barfield A.H.F. equipment can help you.

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Induction heating speeds production

WILD-BARFIELD ELECTRIC FURNACES LIMITED

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### LABORATORY METHODS

MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC

INSTRUMENTS AND MATERIALS

AUGUST, 1961

Vol. LXIV, No. 382

# A Conductimetric Method for the Determination of Carbon and Sulphur in Steel

W. R. Nall and R. Scholev, A.Met., A.I.M.

Bragg Laboratory, Naval Ordnance Inspection Establishment, Sheffield 9.

The determination of carbon (0·1-1·0%) and sulphur (0·005-0·2%) may be made on steel samples weighing 0·05-0·10 g. Alternatively, by substituting a wider combustion tube, the apparatus may be used for the determination of low carbon contents (0·002-0·10%) using a sample weight of 3 g. Both elements are determined after evolving the gaseous oxides by combustion and successive absorption of the gases in different electrolytes. The change in conductivity of the electrolytes is used as a measure of the amount of gas absorbed. The time required for the combined determination is 5-10 minutes.

SEVERAL workers have described conductimetric techniques for the determination of carbon in steel, 1-6 some of which involved complex and expensive apparatus. It was considered that the conductimetric technique would have certain advantages over the gravimetric procedure? if a simple but sufficiently sensitive train could be developed. The G.K.N. Group Research Laboratory demonstrated at the Physical Society Exhibition in 1948 a conductivity cell and meter which had been designed for the determination of small amounts of carbon in silicon-iron alloys, and it was thought that this would be suitable for further investigation and development. A full description of this conductivity cell and meter was made available to the authors.

An apparatus incorporating the main features of the G.K.N. cell and meter design was made and tested on steel samples. During the experiments it was realised that it should be possible to incorporate a conductimetric determination of the sulphur content simultaneously, provided there was no retention of carbon dioxide in the intermediate cell used for sulphur measurement. It was found by careful determination that even large amounts of carbon dioxide, resulting from the combustion of high carbon steels, were quantitatively removed from the sulphur cell. As a result of these experiments it was decided to modify the whole apparatus to allow both elements to be determined conductimetrically.

#### Apparatus

Fig. 1 shows in diagrammatic form the apparatus assembled : the components are as follows :—

- (1) Oxygen cylinder and B.O.R. 12 regulator.
- (2) "Rotameter" type flowmeter.
- (3) Oxygen pre-heater.
- (4) Purification train.
- (5) Platinum-wound combustion furnace.

- (6) Pregl precision pinchcock.
- (7) Sulphur conductivity cell.
- (8) Carbon conductivity cell.
- (9) Conductivity meter.
- (10) Oxygen by-pass.

Oxygen from a cylinder passes first into a "Rotameter" type flow-meter, the rate of flow being controlled at 50 ml./min. The gas is then heated in a furnace tube to a temperature of  $600^{\circ}$  C. in contact with platinised asbestos. Any hydrocarbon impurities present in the oxygen are decomposed to carbon dioxide and water, which are removed by absorption in the next section of the apparatus. Two tubes 12 in. long  $\times \frac{1}{2}$  in. diameter, packed with magnesium perchlorate (anhydrone) and soda asbestos, respectively, are arranged in series, the water being absorbed by the magnesium perchlorate and the carbon dioxide by the soda asbestos. The purified oxygen then passes into the combustion tube. A stopcock A is placed in front of the tube enabling the supply of oxygen to be diverted to the conductivity cells.

The combustion tube is heated in a platinum wirewound furnace incorporating a 12 ft. length of 0.024 in. diameter wire wound on a mandrel giving a central 3 in. hot zone at a temperature of  $1400^{\circ}$  C. The maximum current required is 8 amps at 50 volts A.C. The furnace housing is so constructed that either a Morgan T. 6310 ( $\frac{2}{3}$  in. bore) or 78490 ( $\frac{3}{4}$  in. bore) combustion tube may be used.

The oxygen and the gaseous products of combustion then pass through the precision pinchcock into the sulphur conductivity cell.

Sulphur Conductivity Cell

The cell (Fig. 2) comprises two sections joined together by a standard B29 glass joint. The lower half, the cone of the joint, is the absorption vessel containing the

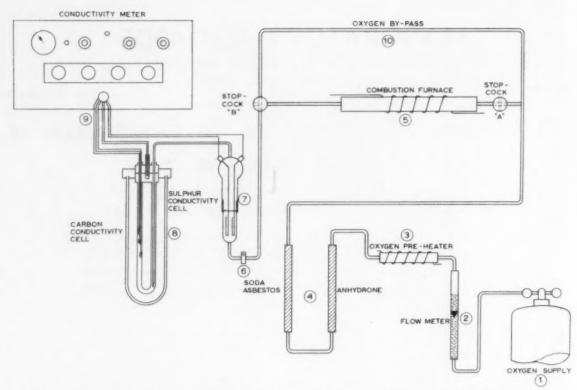
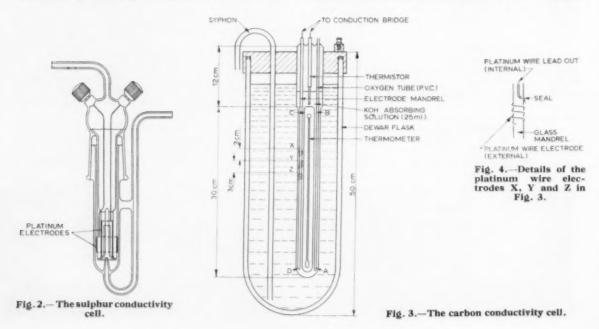


Fig. 1.- Diagrammatic sketch of the equipment for the conductimetric determination of carbon and sulphur in steel.

electrolyte. The gas inlet, in the form of a capillary nozzle, is situated in the base of this section. To the cap is fabricated the electrode assembly, comprising two concentric platinum gauze cylinders between which

conductivity measurements are made. The electrodes are held firmly in position on a glass frame allowing free circulation of the electrolyte. The base of the assembly forms an inverted funnel, which, when the two halves of



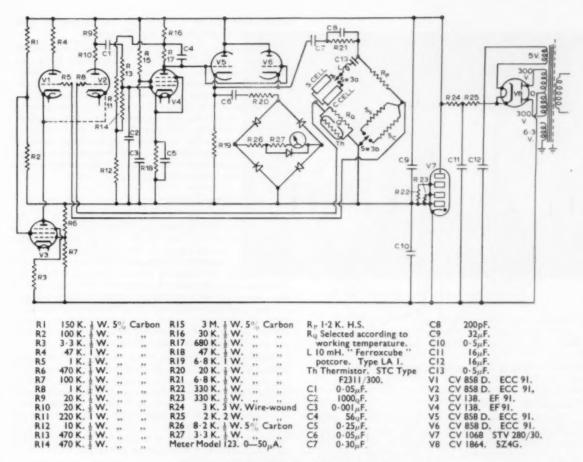


Fig. 5.—Circuit diagram of the equipment.

the cell are in position, fits over the inlet nozzle directing the stream of bubbles up the centre of the inner electrode. This produces efficient stirring of the electrolyte and prevents bubbles from passing between the electrodes, causing erratic electrical measurements. The platinum leads are sheathed in glass and enter the cell through gas-tight seals in the top of the cap. The gas outlet is also situated in the cap allowing the sulphur-free gases to pass into the carbon conductivity cell.

#### Carbon Conductivity Cell

The absorption vessel detailed in Fig. 3 consists of a glass U-tube of 6 mm. bore connected to a cylindrical vessel of internal diameter 25 mm. The electrode system is prefabricated on a glass mandrel prior to insertion in one of the arms of the U-tube. The electrodes are formed by passing lengths of platinum wire down the inside of the mandrel and out through small holes suitably positioned, as shown in Fig. 4. Three turns are wrapped round the outside to give contact with the electrolyte, and the small holes are then sealed off and the wires insulated from each other inside the mandrel. The mandrel tube is filled with wax, ensuring water-tight seals. The outer two electrodes, X and Z, are connected externally and the electrical conductance measured between the inner

electrode Y and the outer electrodes X and Z. This electrode arrangement prevents any current flowing between electrodes X and Z by way of the other arm of the U-tube, where the presence of bubbles would cause erratic results.

The cell is charged with 25 ml, of 0.05 N potassium hydroxide and 0.1 ml. of a 0.1% solution of a non-ionic

wetting agent.

The gases are introduced via a PVC tube at point A and the bubbles rise up the arm A-B, 30 cm. in length, ensuring adequate time for complete absorption of carbon dioxide at a flow rate of 50 ml./min. The presence of the wetting agent in the electrolyte serves a two-fold purpose. The size of the bubbles rising in arm A - B is reduced considerably, presenting a larger absorption surface per unit volume of gas; also a 0.5 in. layer of froth is formed on the surface of the electrolyte, trapping any carbon dioxide which may not have been absorbed. The presence of the bubbles rising in the arm  $A \cdot B$  causes a difference in the hydrostatic pressure between the points A and D, resulting in a rapid pumping action throughout the cell. This ensures that the solution is well stirred and continuously replaced at the electrodes X. Y and Z in the arm C.D. which remains free from

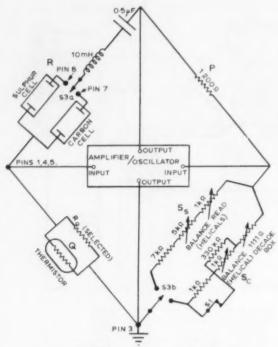


Fig. 6.- Details of the bridge circuit.

#### Temperature Compensation

Electrolytes possess a very large negative temperature coefficient of resistivity and in the case of the carbon determination this presents a possible source of error. In a non-compensated cell a temperature change of 0.5° C. produces a change in conductivity equivalent to 170  $\mu$ g. of carbon, equivalent to 0.017% C on a 1 g. sample. To compensate for this, a thermistor is included in the bridge circuit. Unfortunately, adequate temperature compensation is only afforded over a limited temperature range. The value of this range for a particular thermistor depends upon the shunt resistance  $R_0$  (Fig. 6). The value of this resistance in parallel with the thermistor is selected so that, at the working temperature chosen for the water bath surrounding the carbon conductivity cell, the temperature coefficient of arm Q is equal to that of arm R.

The correct working temperature for a given value of  $R_Q$  is found by plotting a resistance/temperature curve over the range say  $20^\circ$ – $30^\circ$  C. and noting the temperature at which the temperature coefficient is zero. The optimum working temperature can be found by allowing the cell and water bath to attain a steady temperature over a period of several days under normal variations of ambient temperature. This value can then be used for the adjustment of the shunt resistance  $R_Q$  as described above

In the bridge circuit here described the temperature range is  $27 \cdot 5^{\circ} - 28 \cdot 5$  C. To control the temperature of the cell within this range it is immersed in water contained in a Dewar flask 500 mm.  $\times$  100 mm. diameter. A syphon is fitted to the flask enabling it to be emptied rapidly and the temperature of the water adjusted. A

temperature of  $28^{\circ} \pm 0.5^{\circ}$  C. is maintained in this manner for approximately eight hours.

No temperature control or compensation is required in the sulphur cell, as the change in conductivity due to the slight temperature variation which may take place during a single determination is very small in relation to the change in conductivity due to the absorption of the sulphur gases. A change in the bridge resistance of 80 ohms represents approximately 0.010% sulphur. The change in bridge resistance due to a  $0.5^{\circ}$  C. change in cell temperature is 42 ohms, representing 0.005% sulphur. The maximum change in temperature during a single determination under normal laboratory conditions is in the order of  $0.05^{\circ}$  C.

#### The Conductivity Meter

Fig. 5 shows the complete circuit diagram of the apparatus and Fig. 6 illustrates the method of connection of the conductivity cells in the bridge circuit. The alternating current supply to the bridge is derived from a high gain amplifier of zero phase shift, which operates as an oscillator whenever arm S is smaller than the resistance value required for balance. The frequency of the alternating voltage supplied to the bridge is selected by the amplifier/oscillator to produce zero phase shift between input and output, and is such that the arm R acts as a pure resistance giving automatic compensation for any changes in self-capacitance of the cell.

Since power is applied to the bridge only when the bridge is out of balance and when the S arm resistance is too low, it is necessary to operate the bridge with an out-of-balance ratio of a fixed value. The recommended meter reading for "balance" is 10 microamperes, corresponding with a bridge input voltage of approximately  $0\cdot 1$  volt. At this level the arm S can easily be adjusted to the nearest  $0\cdot 2$  ohm, corresponding with approximately  $1\cdot 1$  micrograms of carbon.

The electronic equipment is built onto a chassis within a metal case. The layout of the front panel is indicated in Fig. 7. The top section of the panel contains a microammeter to indicate balance and three helical potentiometers. The "balance carbon" and "read sulphur" potentiometers are graduated in 1 ohm divisions. The centre potentiometer "balance sulphur" is graduated in 5 ohm increments. The switch "carbon-sulphur" is a simple toggle switch permitting either the carbon or the sulphur cell together with the corresponding arm Sc or Ss to be connected to the bridge. The switch operating the 1,000 ohms resistance (S1 in Fig. 6) is located between the microammeter and the "balance carbon potentiometer. The centre of the panel is occupied by a four decade resistance box (" read carbon ") having a total resistance of 1,111 ohms. The 8-pin plug carrying the cell connections is situated in the lower portion of the panel together with an on-off switch, mains fuse and pilot light.

#### **Experimental Work**

The stoichiometry of the carbon dioxide evolution, absorption and conductimetric measurement<sup>1-6</sup> is well established, and the majority of the experimental work has been concerned with the conductimetric determination of sulphur. The two principle factors to be considered were (i) the selection of a suitable electrolyte which would absorb the sulphur gases with a large change

in conductivity, and (ii) the design of a cell capable of measuring this change.

The choice of electrolyte lay between a number of solutions of oxidising agents such as hydrogen peroxide, potassium dichromate, potassium permanganate, thallic chloride and ceric sulphate, which all react with sulphur dioxide to form either sulphuric acid or a sulphate. A second important feature of the electrolyte was that no reaction must take place between it and either carbon dioxide or oxygen. Koch, Eckhard and Malissa<sup>6</sup> used acidified potassium dichromate and suggested that potassium permanganate and hydrogen peroxide were unsuitable.

To obtain a large change in conductivity for a small increase in sulphuric acid or sulphate concentration, the initial conductivity k must be small. k=l/AR, where l is the distance

between the electrodes, A is the area of the electrodes, and R is the resistance between them. As R decreases k increases with a given electrode arrangement. If strong salt solutions are used the initial resistance between the electrodes will be small and the conductivity, k, will be large.

Of the oxidising agents previously mentioned, hydrogen peroxide appeared to be the most suitable from the point of view of obtaining an initial low conductivity. The electrode assembly was designed with this in mind. The distance l was kept to a practical minimum and the area of the electrodes A was made as large as possible.

Platinum acts as a catalyst in the decomposition of hydrogen peroxide and early trials were not satisfactory as bubbles of gas, covering the surface of the electrodes, completely altered their characteristics. Other electrolytes were tried in an attempt to overcome this, but the initial high conductivity resulted in a loss of sensitivity. Ceric sulphate for example, produced such a low cell resistance that it was beyond the range of the meter, contamination of the electrodes occurred in the presence of potassium permanganate, and, as thallic chloride was not available commercially, the only remaining salt solution was potassium dichromate. Successful tests with the latter were carried out using a 0.01% solution acidified to pH 3 with hydrochloric acid. as suggested by Koch et ale, but the sensitivity was much less than with hydrogen peroxide.

Previous attempts have been made to suppress the catalytic action of platinum upon the decomposition of hydrogen peroxide. The presence of a thin film of platinum oxide appears to have a suppressive action, and by allowing the electrodes to stand immersed in strong (100 vol.) hydrogen peroxide for at least 24 hours it is thought that such a film is produced. After this treatment no decomposition of the peroxide was noted in the cell. Dilute phosphoric acid was added (0·005%) as an extra stabilising agent for the hydrogen peroxide (0·10 vol.).

The ratio of sulphur dioxide to sulphur trioxide and the total amount of sulphur gases evolved from the steel

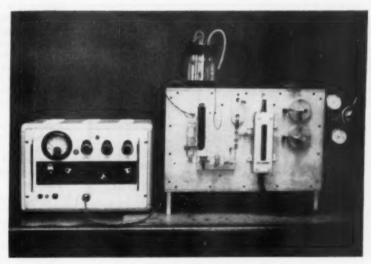


Fig. 7. General view of the equipment.

on ignition depend upon the conditions prevailing in the furnace. The rate of oxygen flow, the temperature of the combustion tube, and, most important, the fluidity of the slag after combustion, all have an effect upon the yield of oxides of sulphur for a given sulphur content of the sample. The rate of oxygen flow and the combustion tube temperature are easily controlled at 50 mL/min. and 1,350° C., respectively, but the fluidity of the slag will depend upon the type of steel being analysed. Some steels, notably alloy steels, form a viscous slag which tends to trap gases in the bubbles formed. The addition of pure tin granules removes this source of error by increasing the fluidity of the slags and enabling the gases to escape freely.

#### Method

#### Preparation of Sample

The sample should be prepared in a sufficiently fine state of division to obtain results representative of the composition of the material as a whole. This is effected by one of the following methods, depending on the shape and dimensions of the sample and whether all the sample or select portions are required for analysis.

- (a) Machining in a milling machine. This gives very accurate control when shallow, adjacent steps have to be obtained over a small distance and the millings collected separately.
- (b) Turning on a small lathe.
- (c) Drilling with a very fine drill.
- (d) Flattening in a hardened steel mortar followed by cutting into small pieces with shears. This treatment has been found useful with materials such as stainless steel wires which will not burn off if left in the form of wire. The efficiency and completeness of combustion increases as the sample thickness is reduced.

The sample is then examined with the aid of a magnifying lens and any obvious impurities are separated, using fine forceps. Non-magnetic impurities are separated by placing the sample between two sheets of glossy

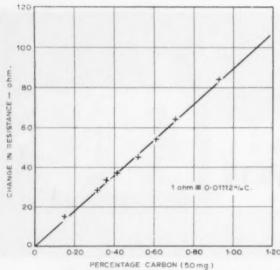


Fig. 8.—Calibration curve for the conductimetric determination of carbon.

paper and attracting the steel to the upper sheet with a powerful magnet. The non-magnetic portion which stays behind on the lower sheet may then be removed. Any oil or grease contamination is removed by washing the sample several times with diethyl ether and drying at a temperature between  $180^\circ$  and  $300^\circ$  C. This ensures that the organic solvent is completely removed from the metal surface. The sample is then stored in a stoppered glass specimen tube.

#### Procedure

Switch on the furnace and increase the temperature gradually to 1,350° C. by means of the variable resistance. Adjust the oxygen supply pressure to 1 lb./sq. in. using a B.O.R. 12 regulator and adjust the flow through the apparatus to 50 ml./min. by means of the precision pinchcock. Fill the Dewar vessel surrounding the carbon conductivity cell with water at the correct working temperature and check the conductivity meter for the following points:—

- (a) The "carbon-sulphur" changeover switch is in the "carbon" position.
- (b) The "balance carbon" potentiometer reads 1,000 ohms and the 1K resistance switch is in the open position.
- (c) The "read carbon" decade reads maximum.
- (d) The "balance sulphur" potentiometer reads 5,000 ohms.
- 5,000 ohms.
  (e) The "read sulphur" potentiometer reads zero.

Switch on the meter, remove the PVC gas delivery tube from the carbon cell and charge with 25 ml. of 0.05 N potassium hydroxide solution and 0.1 ml. of a 0.1% solution of a non-ionic wetting agent. Separate the two halves of the sulphur cell and, with the oxygen flowing, transfer 15 ml. of 0.1 vol. hydrogen peroxide in 0.005% phosphoric acid to the lower half of the cell. Carefully replace the upper section of the cell, ensuring a tight fit at the joint. Slowly lower the PVC inlet tube down the arm of the carbon cell, maintaining a con-

tinuous flow of bubbles. Allow oxygen to flow through the apparatus for at least 1 hour.

Rotate the stopcocks B then A, allowing the oxygen to pass through the by-pass, and remove the glass tube closure from the end of the combustion tube. Place a few milligrams of steel in a combustion boat and insert into the hot zone of the tube. Replace the tube closure and rotate the stopcocks A then B, allowing the oxygen to pass through the combustion tube. After 10 minutes adjust the "balance carbon" potentiometer until a reading of 10 micro-amperes is observed on the meter. Move the changeover switch to the "sulphur" position and adjust the "balance sulphur" potentiometer until a reading of 10 micro-amperes is again observed on the meter. Replace the change-over switch to the "carbon" position.

Prepare the sample as described above and transfer the appropriate weight to a combustion boat (Note 1). Cover the sample in the boat with pure tin granules. Rotate the stopcocks B then A, allowing the oxygen to pass through the by-pass, and remove the tube closure. Withdraw the used boat from the tube and place the sample boat in the hot zone of the furnace. Replace the tube closure and rotate the stopcocks A then B. allowing the oxygen to pass through the combustion tube for 10 minutes. Adjust the dials of the "read carbon" decade box until a reading of 10 micro-amperes is observed and note the decade reading. Rotate the "read sulphur potentiometer until at least 500 ohms is shown on the dial (Note 2), move the changeover switch to "sulphur' and adjust the dial back until a reading of 10 microamperes is shown. Note the reading on the dial.

Relate the readings of both the "read carbon" decade box and the "read sulphur" potentiometer to pre-determined calibration graphs to obtain the percentage carbon and sulphur of the sample. A blank determination on the boat, flux and oxygen should be made in the following manner.

When a steady reading is being recorded on the microammeter with oxygen passing through the combustion tube, place the required amount of pure tin granules into the appropiate combustion boat. Operate the stopcocks B and A and remove the tube closure from the end of the combustion tube as described above. Place the combustion boat in the hot zone of the furnace tube and allow the oxygen to pass. Note the change in resistance required to balance both for carbon and sulphur.

Deduct the blank readings thus obtained from readings recorded on samples. Blank readings of the following order are obtained.

Sample Weight	Change in Resistance	Equivalent Carbon
0.05 g.	0.5 ohm.	0.008%
3 g.	$2 \cdot 0$ ohm.	0.0005%

Blanks recorded on the sulphur determination are less than 5 ohms, representing less than 0.0005% sulphur. Notes

NOTE 1

Sample Weight	Boat Size	Carbon Content
0.05-0.10  g.	"OO" $(30 \times 6 \times 4 \text{ mm.})$	

The combustion boats referred to above may be obtained from Messrs. Alpolain Industries, Ltd., 5 Dalewood Road, Sheffield 8.

#### NOTE 2

If the changeover switch is operated without any prior increase in resistance, then a relatively high current will flow through the meter. The quantity of heat thus dissipated in the thermistor will be sufficient to cause a false temperature compensation, involving an erroneous result. To prevent this, the meter reading must be kept below 20 microamperes; if this figure is exceeded, two or three minutes must be allowed for the thermistor to regain its correct temperature.

#### Calibration

#### Carbon

The following three methods are available:-

(i) Transfer 25 ml. 0·05 N potassium hydroxide to the cell and add accurate aliquots of 0·05 N potassium carbonate solution, noting the balance conditions for each addition. The change in readings on the "read carbon" decade box represents the change in resistance required in the bridge arm S to balance the increase in resistance of the cell due to the addition of potassium carbonate.

The carbon content of each 1 ml. portion of  $0\cdot05$  N potassium carbonate solution is  $0\cdot3$  mg., but since the determination of carbon by evolution would normally maintain the cell volume constant at 25 ml., the concentration of carbon per 25 ml. of electrolyte does not increase by  $0\cdot3$  mg. per ml. of potassium carbonate added, but at a reduced rate represented by the equation :

mg. of carbon = 
$$\frac{7 \cdot 5 \, n}{25 + n}$$

where n is the number of millilitres of 0.05 N potassium carbonate solution added.

The change in readings on the "read carbon" decade box when plotted against the theoretical amount of carbon added gives a straight line calibration graph.

TABLE I.—RESULTS OBTAINED USING THE CONDUCTIMETRIC TECHNIQUE.

Sample B.C.S.* Weight No.	0.000	Carbon P	ercentage	Sulphur Percentage		
	Standard Figure	Results	Standard Figure	Results		
50 mg. 914 239/1 218/1 225 224 232 159/1 152/1	214	0-375	0·38 0·38	0.033	0-039 0-034	
	239/1	0-32	0-325 0-315	0.025	0 · 024 0 · 025	
	218/1	0-155	0-16 0-155	0-045	0-043 0-046	
	0-37	0-365 0-37	0-016	0-014 0-015		
	0.29	0·385 0·39	0.029	0·029 0·030		
	0.20	0 · 205 0 · 21	0-067	0.067 0.067		
	0-52	0-51 0-515	0.045	0-043 0-045		
	0.07	0.08 0.085	0-259	0·24 0·26		
1 g. 264 265 150	0-037	0-037 0-037	-	-		
	0-047	0.048 0.049	- 1	-		
	0-029	0·029 0·029	-	-		
3 g.	149/1	0.003	0-0028 0-0027	-	-	

British Chemical Standard.

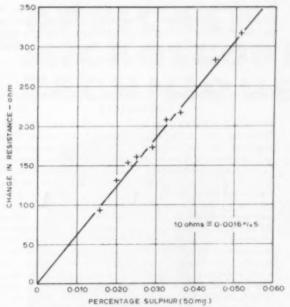


Fig. 9.—Calibration curve for the conductimetric determination of sulphur.

- (ii) Ignite weighed amounts of dry calcium carbonate in the apparatus and plot the theoretical carbon content against the change in resistance measured on the "read carbon" decade box.
- (iii) Ignite a series of standard steels in the apparatus. This enables the simultaneous calibration for carbon and sulphur to be made.

A calibration graph obtained by method (ii) and checked by method (iii) with appropriate blank correction is shown in Fig. 8.

#### Sulphur

The only method at present available for the calibration of the sulphur determination is to ignite a series of steels of known sulphur content under the standardised conditions of the method. The temperature of the furnace must be  $1,350^{\circ}$  C. and the rate of oxygen flow 50 ml./min. T. B. Smith et al\* found that under optimum conditions of temperature and flow rate the maximum percentage of sulphur evolved is 94% of the total amount present in the sample. A graph relating change in resistance, as measured on the "read sulphur" potentiometer, with known percentage sulphur in the steel shows a straight line relationship. (Fig. 9.)

#### Results

Results obtained using the above technique compare favourably with those given by other methods using a similar sample weight. Typical results are shown in Table I.

#### Acknowledgment

The co-operation of Mr. G. Croall, head of the chemistry department, G.K.N. Research Laboratories

in supplying details of the carbon cell and meter is gratefully acknowledged. Our thanks are also due to Mr. W. N. Smith of the Bragg Laboratory for the help given in constructing the electronic equipment This paper is published by permission of the Admiralty.

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#### Copper Roofing Screens New Research Laboratory

RUBEROID COPPER ROOFING, manufactured by The Ruberoid Co., Ltd., has been used to screen from electrical interference one of the new pharmacological and biochemical laboratories at the Beecham Research Laboratories, Betchworth, Surrey. The laboratory is being used for electro-physiological experiments—such as measuring the potential in nerve fibres—which would be vitiated by outside interference. For this purpose it was necessary to sheath all the internal surfaces, ceiling, walls and floor, with a good conductor in continuous electrical contact at earth potential, and to ensure that the multiple service pipes or conduit were isolated from outside interference. Ruberoid copper roofing was selected because it proved to be more pliable, and there was a considerable saving in cost compared with plain copper sheeting. Fixed by the normal adhesive method, it was then anchored with nails to the timber battens set into the walls and ceiling. Copper strips were soldered after fixing over all the joints to achieve electrical continuity.

Copper-faced internal shutters screen the window and door openings under actual test conditions, and a plenum air conditioning system supplies and extracts fresh air through ceiling vents, which are screened with fine copper gauze. The architect, Mr. F. Senior, and the scientists concerned, have expressed their complete satisfaction with the results. The main contractors were Percy Bilton, Ltd.: sub-contractors for the taping and earthing required to complete the installation were Buchanan and Curwen, Ltd., who also developed the method used for rapid soldering of the copper strips.

This unusual application of Ruberoid copper roofing was carried out by the contract division of The Ruberoid Co., Ltd., in close collaboration with the main contractors. Ruberoid copper roofing is extremely durable and suitable for use on any type of building, where it is advantageous to secure the weatherproofing qualities of bituminous roofing coupled with the appearance of a copper roof.

#### Hilger & Watts in Poland

"How long does an analysis take?" "Where are your instruments working?" "How many of your staff are engaged on research?" These are typical of the ques-"How many of your staff are tions answered by Mr. Hamill of Hilger & Watts. Ltd... at the end of a lecture on automatic spectrographs, given to sixty-five representatives of the steel industry in Catowice. Upper Silesia, in early June. A large automatic Hilger spectrograph is already installed and is giving very good results in Warsaw, and local authorities were very helpful in arranging the lecture, which was given by Herr Dipl. Ing. Herbert Eischer.

All the automatic spectrographs made by Hilger & Watts were discussed, but the one of particular interest to the steel industry was the Polyvac, which analyses up

to nineteen elements and types out the results in less than three minutes; the instrument deals with carbon, phosphorus, sulphur, silicon, and any metallic element. In the same week two demonstrations were arranged for metallurgists in Catowice and Warsaw. These showed the Hilger atomic-absorption equipment, used with the Uvispek spectrophotometer for the analysis of metals in solution. There are many advantages in this method of analysis-the accuracy is largely unaffected by flame temperature, there is little or no inter-element interference, and generally a single set of standards is sufficient.

#### Griffin-Grundy Furniture Link

GRIFFIN & GEORGE, LTD., and Grundy Equipment, Ltd., have been associated for several years in the manufacture and marketing of metal laboratory furniture and laboratory apparatus, including service racks, Griffin-Grundy ovens, Grundy solenoid valves, etc. A still closer link has now been forged whereby the Grundy group becomes responsible also for the manufacture of wooden laboratory furniture. Marketing will continue to be handled solely by Griffin & George (Laboratory Construction), Ltd., whose sales department is operating from High Street, Cowley, Uxbridge, Middlesex,

#### **News in Brief**

SOUTHERN ANALYTICAL, LTD., have been participating in the exhibition section of the 5th International Congress of Biochemistry in Moscow, 10th to 16th August, where they showed a wide range of polarographic equipment including the Southern-Harwell pulse polarograph and Davis differential cathode ray polarograph together with other instruments of interest to the analyst.

FIELDEN ELECTRONICS, LTD., have announced the opening of a fully equipped service department in the building housing their Walsall offices.

ALUMINUM CO. OF CANADA, LTD., is to double its capacity to roll aluminium sheet in Canada in the next 18 months through the construction of a new modern sheet mill at Kingston, Ontario. The new mill will be capable of rolling wider sheet and plate and larger coils of sheet than have hitherto been available from Canadian

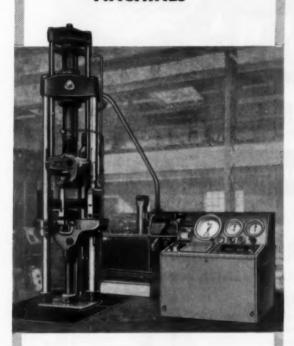
THE General Electric Co., Ltd., of England has recently received an order worth approximately £125,000, for a materials handling plant with a maximum unloading capacity of 2,000 tons/hr., to be installed in a self-unloading ship. The order was received from William Hamilton and Co., Ltd., of Glasgow, who are building the vessel for North American owners; when the ship is commissioned it will operate in Canadian waters.

The address of John Miles & Partners (London), Ltd., consulting engineers, is now Moor House, London Wall, London E.C.2. (Telephone: METropolitan 0471; telegraphic address: MILESCO, STOCK, LONDON.)



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broad and practical approach adopted in this book, the emphasis placed on technique, and the number of references provided will render it of value to users of the optical microscope in various fields. Since it is a practical work, intended for reference at the bench, each section has deliberately been made as self-contained as possible; while this naturally entails some repetition it is believed that the advantages gained by the arrangement outweigh the disadvantages. The first chapter deals with the principles of microscopy and describes some of the characteristics and limitations of microscope equipment. In the next chapter the application of these principles in modern equipment is discussed, with the aim of showing something of the range of general purpose and specialized equipment now available. Simple bench microscope technique is dealt with in some detail in chapter free, a section which it is hoped will be of particular interest to the student. Specialized techniques of illumination and observation are discussed in chapter four, with particular emphasis on the potentialities and limitations of various techniques. The preparation of transparent and opaque specimens is covered in chapters five and six, and the final chapter deals with photomicrography. The aim throughout has been to emphasize the basic principles involved.

CONTENTS

#### CONTENTS

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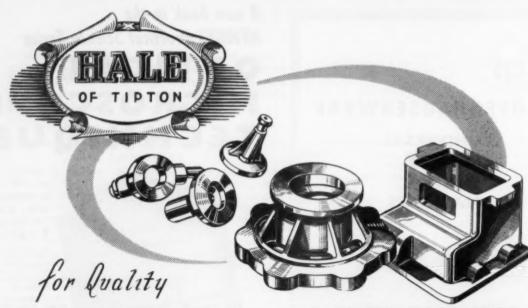
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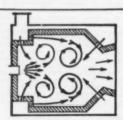


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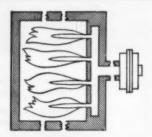
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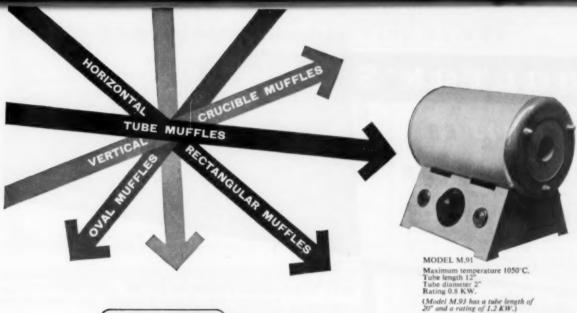
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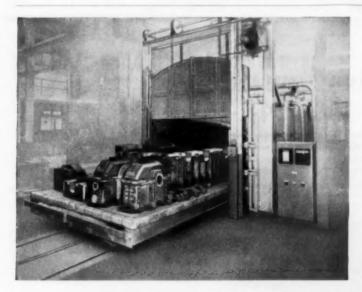


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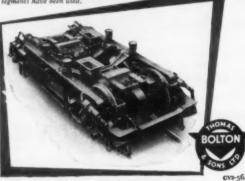




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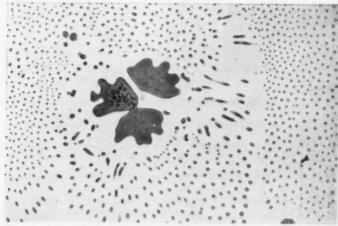
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